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Fiber Reinforced Solids Possessing Great Fracture Toughness: The Role of Interfacial Strength

Final Technical Report

Extension of
NASA Grant
NGR 23-005-528

August 1, 1974 - July 31, 1975

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INTERMITTENT BONDING
FOR
HIGH TOUGHNESS/HIGH STRENGTH
COMPOSITES

BY

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This report is predicated, in some degree, upon knowledge contained in the Final Report on Grant NGR 23-005-528, July 31, 1974.

Acknowledgements

Dr. Buck Fichter, of the NASA Langley Research Centre, has been a most helpful monitor of this grant and I am pleased to acknowledge his cooperation, and also that of his colleagues. Thanks are also owed to Dr. Yiu-Wing Mai and Mr. William Durrant for much of the experimentation, particularly the recent environmental studies, and to Mr. Robert Cisler who filled the indispensable role of chief tape-maker.

List of Symbols and Abbreviations

A_{nom}	Nominal cross sectional area of test-specimen in path of crack
C	Coating fraction (decimal l_c/l_r).
D	Average distance of fractured filament from plane of gross fracture.
d	filament diameter
E	Young's modulus
H	Height of edge crack testpiece arms
h	Pulled-out relative slip distance
K	Stress intensity factor
L	Finite size of testpiece or length of discontinuous filament
l	Length
N	Number of filaments
n	Ratio of finite length of filament to repeat length ($=L/l_r$)
PUV	Polyurethane varnish
R	Fracture toughness
RoM	'Rule of mixtures'
SVG	Silicone vacuum grease
T	Ratio of coated to uncoated interfacial shear strengths
v	Volume fraction
λ	Ratio of repeat length to filament diameter
ρ	Ratio of coated toughness to uncoated toughness
σ	Tensile stress
τ	Shear Stress
ψ	Ratio of uncoated critical length to repeat length, $(l_{crit})_{uc}/l_r$.

Superscripts

τ' Interfacial shear stress during pull-out

Subscripts

I,II	Fracture toughness crack opening modes
av	Average
c	Coated
crit	Critical
f	Filament
if	Interface
m	Matrix
r	Repeat length
uc	Uncoated

DATA SHEET

Filaments:

$$\sigma_f = 3.45 \text{ GN/m}^2$$

$$E_f = 380 \text{ GN/m}^2$$

$$d = 140 \text{ } \mu\text{m}$$

Matrix:

$$\sigma_m = 81 \text{ MN/m}^2$$

$$E_m = 2.48 \text{ GN/m}^2$$

$$R_m \approx 2-3 \text{ kJ/m}^2$$

$$\tau_m \approx 69 \text{ MN/m}^2$$

Composites:

$$(l_{crit})_{uc} = \frac{\sigma_f d}{2\tau} \rightarrow 3.5 \text{ mm}$$

$$\psi = \frac{(l_{crit})_{uc}}{l_r} \rightarrow \begin{cases} 0.18 & \text{for } l_r = 19 \text{ mm} \\ 0.14 & \text{for } l_r = 25 \text{ mm} \\ 0.07 & \text{for } l_r = 51 \text{ mm} \end{cases}$$

$$\lambda = \frac{l_r}{d} \rightarrow \begin{cases} 136 & \text{for } l_r = 19 \text{ mm} \\ 181 & \text{for } l_r = 25 \text{ mm} \\ 363 & \text{for } l_r = 51 \text{ mm} \end{cases}$$

Part I

Angle Ply Results

LARGE FRACTURE TOUGHNESS BORON-EPOXY COMPOSITES

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In conventional brittle fiber/brittle filament composites, when the interfacial bond between filament and matrix is strong, fracture is often caused by rapid matrix cracks which break through all filaments in their paths. The toughness of such composites is low because, in general, the critical transfer length associated with strong interfacial bonding is small, which limits the various components of the total toughness—the 'surfaces' component, Piggott-Fitz-Randolph stress redistribution and Cottrell-Kelly pull-out (see, for example, ref. 1). The critical length is given by $l_{crit} = \sigma_f d / 2\tau$, where σ_f is the filament strength, d the filament diameter, and τ the interfacial shear strength. A general increase of l_{crit} by lowering the filament-matrix shear bond will increase the toughness, but weak interfaces throughout the composite reduce the tensile strength quite significantly.

Recent work^{2,3} has shown that "intermittent bonding" allows high toughness to be obtained in brittle filament/brittle matrix composites without significant loss of tensile strength. Filaments are arranged to have alternate bands of high and low shear stress (and low and high toughness) by interrupted coating along the filaments with appropriate substances. The strong regions ensure that the filament strength is picked up; randomly positioned weak areas effectively blunt cracks by the Cook-Gordon mechanism⁴ which in turn produces long pull-out lengths with an associated large contribution to toughness. Unidirectional boron-epoxy composites of volume fraction

Index Categories: Materials, Properties of; Structural Composite Materials
(including coatings);

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0.20-0.25 have been made in this way; they have fracture toughnesses of over 200 kJ/m^2 , (as opposed to about 40 kJ/m^2 with no coating), and they retain rule of mixtures tensile strengths ($\sim 650 \text{ MN/m}^2$). At the volume fractions used, that apparently represents K_{IC} values greater than $100 \text{ MN/m}^{-3/2}$, where K_{IC} is the critical stress intensity factor.

Two different coating materials have been investigated^{2,3}, viz: silicone vacuum grease (SVG) and polyurethane varnish (PUV). Both appear to produce similar interfacial shear strengths in the coated regions, since the tensile strength variations with coating geometry are indistinguishable. Their effects on toughness, however, are significantly different: SVG produces only a modest increase in toughness (up to say 60 kJ/m^2 as opposed to some 40 kJ/m^2 when uncoated), whereas PUV coatings give the previously quoted values of over 200 kJ/m^2 . The difference is explained in terms of the occurrence of Cook-Gordon debonding with PUV, but its absence in SVG systems. Thus it seems that interfacial toughness, rather than the commonly considered interfacial shear strength, may be an important parameter controlling overall composite toughness. The relationships between interfacial toughnesses, in various debonding modes, and interfacial strengths are not clear.

This present paper reports the results of recent experiments with angle-ply layups rather than unidirectional composites. The absolute values of strength and toughness are of course lower, but in general terms the same trends are observed, with markedly improved toughness at high percentage PUV coatings.

Tensile strength and toughness specimens were made from layers of intermittently bonded, epoxy composite tape, manufactured on a drum apparatus with a device for coating the filaments, before layup,³ with various coating/uncoating sequences. The tape (similar to Avco Rigidite, Prepreg

tape) consisted of a 250 μm monolayer of B/W filaments in EPON 828 epoxy, backed, for ease of handling, on 760 mm wide nylon scrim cloth about 50 μm thick. Tensile specimens consisted of 5 layers of tape, arranged in the following orientation sequence $0^\circ/+45^\circ/0^\circ/-45^\circ/0^\circ$, where 0° is the pulling direction. The specimens had a gauge section of some 60 mm x 6 mm x 1 mm. The toughness specimens were 7 layer flat coupon specimens (about 76 mm x 76 mm) with long starter cracks, akin to ASTM compact tension specimens⁵ in profile. To prevent the composite arms above the crack from shearing off under load, an additional layer of tape was added to each side of the specimen, with filaments parallel to the crack. The central cores of the specimens thus consisted of 5 oriented filament layers in the path of the crack, where, within the limitations of the specimen and tape preparation method, the coated and uncoated layers occurred randomly relative to each filament. The starter crack in these edge crack specimens was made with a profiled diamond slitting wheel. Toughness was measured for increments of crack area, using Gurney's segmental area technique⁶.

Tensile strength and fracture toughness for the PUV coated crossply specimens are plotted in Figures 1 and 2 against C, the coated fraction.

$C = \ell_c / (\ell_c + \ell_{uc})$ where ℓ_c is the coated length, ℓ_{uc} the uncoated length and $(\ell_c + \ell_{uc})$ the repeat distance of the coating pattern along the filaments.

The promising results obtained earlier for unidirectional filaments have been duplicated in angle-ply layups. The absolute values of strength and toughness are less, of course, for the same volume fraction of filaments. Even so, quite respectable strengths and toughnesses are still obtained (300~400 MN/m² and 100~200 kJ/m² at the higher percentage coatings) coupled with less anisotropy. There is some scatter in the results (cf. the fracture toughness at $C = 0.25$), but the trend is undoubtedly towards greater

toughnesses at high coated fractions.

A significant contributor to total toughness is the pull-out work performed by those filaments bridging the crack face after passage of the main crack front. Such filaments, broken at distances removed from the main crack plane, can stabilize cracking in specimens that have bad geometric stability factors⁷ and which would normally be unstable (e.g. the single edge notch (SEN) tensile specimen). Testpieces with filaments bridging the crack faces will not return to the origin upon unloading because of geometric interference (i.e. the filaments would have to be pushed back up the holes down which they had been pulled). Usually, when unloading lines do not go back to the origin, it is an indication of generalised yielding at regions remote from the crack tip (cf. the difficulties of testing metals, such as low carbon steels, which have large toughness-to-yield-strength ratios). A means of establishing whether gross irreversibilities away from the crack tip have occurred in filamentary composites is to cut through the fibers bridging the crack faces and beyond into virgin material ahead of the crack tip. If the crack then closes up, it may be assumed that the cause of remaining open was simply geometric interference on the part of the filaments, akin to a residual elastic opening moment at the crack tip. Such an investigation of displacement reversibility is important, since all fracture mechanics is predicated on this fact.

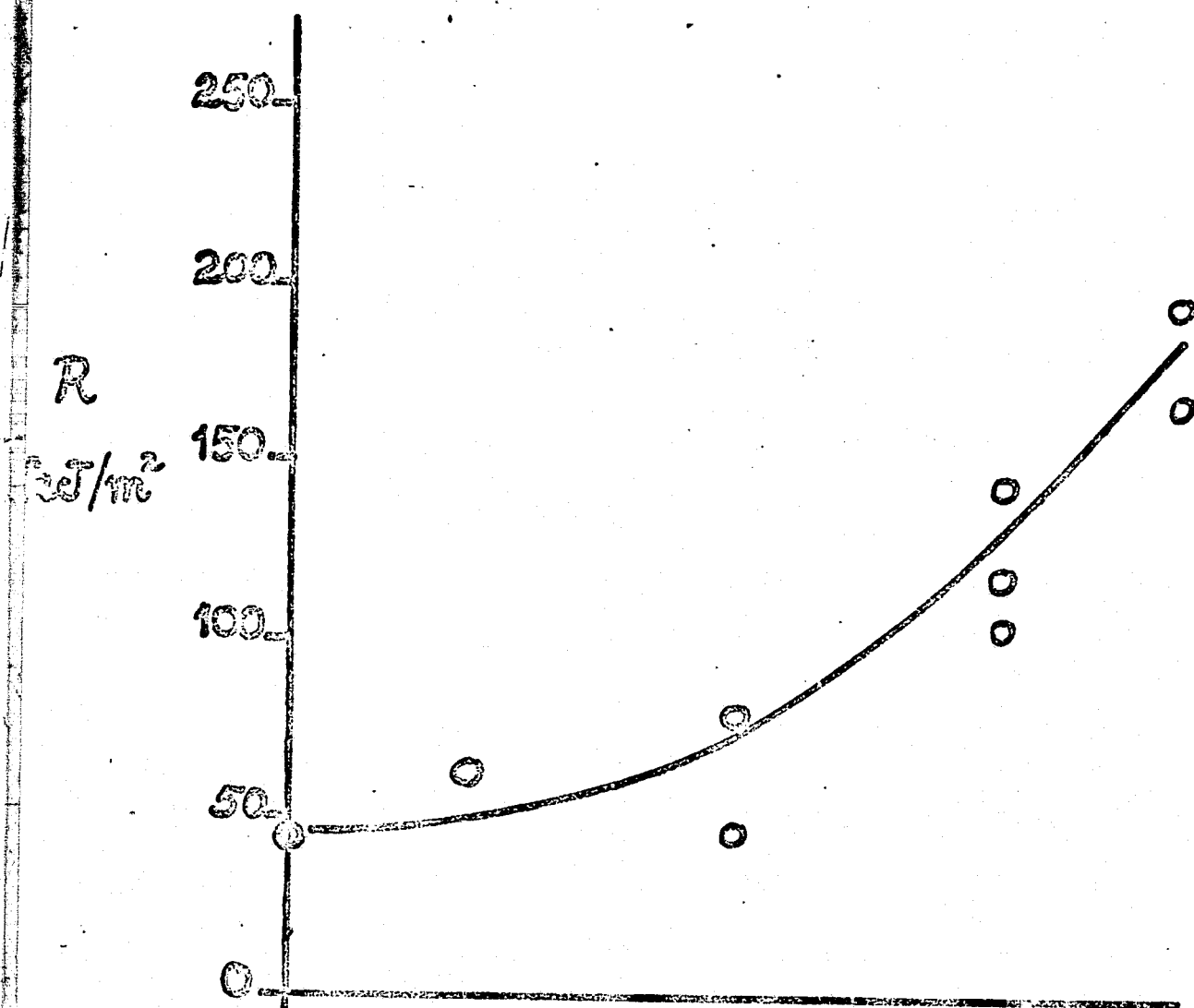
I am pleased to thank NASA for a grant (NGR 23.005.528) under which most of this work was carried out.

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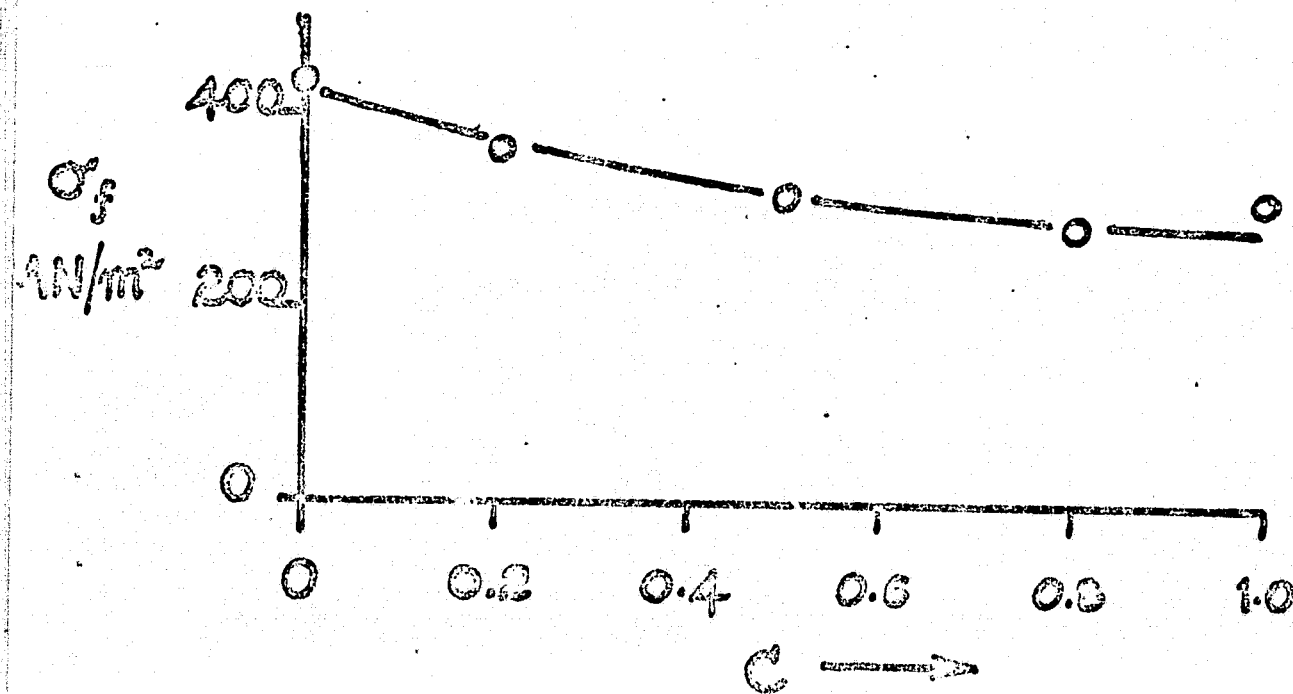
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Figure Captions

1. Fracture toughness of 0.2 volume fraction boron/epoxy "compact tension" profile testpieces versus polyurethane varnish coated fraction.
2. Tensile strength of 0.2 volume fraction boron/epoxy specimens versus polyurethane varnish coated fraction.



Atkin
Fig 1



Atkin
Fig 2

Part II

Effect of Water and Ice on Strength and Toughness

1. Introduction

It was decided to perform an exploratory study upon the effects of environments of water (both liquid and frozen) on the strength and toughness of both unidirectional and angle ply laminates. In any real-life application of intermittently bonded composites, the effects of rain, soaking by water, and possible freezing of that water seem areas that are essential to investigate. It seemed prudent to determine whether all the advantages of intermittent bonding would be retained when wet.

2. Testpieces and Experiments

The same types of specimens were used in this investigation as in the angle ply study, i.e. 5-layer tensile bars, about 115 mm long by 10 mm wide, and 7-layer compact tension profile toughness specimens, 75 mm x 75 mm with a 54 mm starter crack (of which the two outside layers, parallel to the starter crack, were intended to encourage the crack to propagate in a straight line). Both unidirectional (5 parallel layers) and angle-ply (layers at 0, -45, 0, +45, 0) laminates were made for tensile and toughness measurements. All specimens were made from 0.20 ~ 0.25 volume fraction tapes, intermittently bonded with polyurethane varnish to produce the following coated fractions: (a) zero, (b) $C = 0.2$, (c) $C = 0.5$, (d) $C = 0.8$, and (e) $C = 1.0$.

All specimens were submerged in a tray of water and allowed to soak for many days. Every testpiece was removed from the water and weighed at the end of each day, (excess water being lightly removed with blotting paper). The absorption isotherms thereby produced are shown in Fig. 1, where the ordinate represents the percentage gain in weight (measured in grammes/gramme) against number of days. It will be observed that the uncoated specimens took up considerably more water than the others, and in broadest terms, the absorption seems to vary inversely with the coated fraction—although in truth the differences between the data for $C = 0.2, 0.5, 0.8$ and 1.0 are not marked. Later we will

use an argument in the analysis of the strength and toughness results that depends upon the postulate that those interfacial regions coated by polyurethane varnish are unaffected by water, whereas the uncoated areas are significantly affected.

Even though arguably the specimens were still absorbing water after a fortnight, and thus were not fully saturated, it was decided to test them in that wet condition. A number of specimens were set aside, put into the freezing compartment of a refrigerator, and left overnight. Having unthawed, they were put into the programme of tensile and toughness measurements. The experimental details followed previous practice (in particular, fracture toughness was determined by Gurney's irreversible work area method).

3. Results

Figure 2(a) shows the tensile strength results for wet and thawed unidirectional filament testpieces, and Figure 2(b) gives the corresponding data for angle-ply laminates. The results for identical dry specimens (earlier work) are also shown*. There is a marked reduction in the strength of wet uncoated samples ($C = 0$), when compared with the strength of dry uncoated testpieces. On the other hand, there is hardly any difference between the strength of wet and dry fully coated samples ($C = 1$). At intermediate values of C , the data blend in between the $C = 0$ and $C = 1$ results,—the dry specimens falling off in strength between $C = 0$ and $C = 1$, (as discussed and explained in earlier reports), and the wet specimens increasing in strength as the fully coated condition is approached.

The reduction in uncoated wet strength for the angle-ply specimens is not proportionately as great as that of the unidirectional specimens, (about 250 MN/m^2 compared with 450 MN/m^2 , as opposed to about 250 MN/m^2 compared with some

* The volume fractions of the dry samples are closer to 0.2 than 0.25, whereas the present results have v_f closer to 0.25 than 0.2. This affects one-to-one comparisons.

620 MN/m²). This is presumably because the load carried by the 45° angle plies is a smaller proportion of the load carried by the unidirectional filaments, so that the reduced interfacial strength does not produce as big an effect. In the experiments, the uniaxial fibers broke first, then upon load transfer, the 45° filaments broke across themselves.

In all cases in general, the effect of freezing and thawing seems to deteriorate further the reduction in strength when compared with the corresponding dry cases (the 'ice' data point at $C = 0.2$ in Figure 3(a) is dubious).

Figure 3(a) and (b) show the toughness results for unidirectional and angle-ply laminates respectively. Again, data for corresponding dry samples are given, noting (as for the tensile results) that the volume fractions may be somewhat different (see previous footnote). In general, the limited data suggest that toughness levels are not markedly different from the dry samples, except for the highest percentage coating, when the gain in toughness with increasing C is rather limited. Unfortunately many of the specimens failed by crack propagation perpendicular to the starter crack direction. Because of the limited number of test-pieces, it was not possible to recheck some of those conditions. However, even if the toughness results are somewhat imprecise, the evidence seems to be that quite respectable toughnesses are attainable in coated samples even when fully wet, and even after one freezing/thaw cycle.

As in the earlier studies, pull-out experiments (after the filaments had broken) were performed in order to establish the interfacial frictional stress, acting after complete debonding. Specimens were regripped above the remaining crack ligaments and the work estimated from the area under the load/displacement plot on the tensile testing machine. As shown in the photographs, many filaments pulled out over long lengths, which made it very difficult to count individual filaments. To bypass this problem, the number of filaments pulling out was estimated from the known volume fraction of the specimens, and the remaining

crack ligament over which pull-out was taking place.

In this way, the following average values of the interfacial frictional stress during pull-out were established.

σ_c	pull-out τ' friction (MN/m ²)	Condition
0	0.49	water soaked
0.2	0.41	"
0.5	0.46	"
1.0	2.75	"
0	0.12	frozen & thawed
0.5	1.54	"

These values should be contrasted with $\tau' \approx 2 \text{ MN/m}^2$ from earlier "dry" tests.

4. Analysis of Results

4.1 Interfacial Shear Strengths

The tensile strength results seem to suggest that the water (and ice) affected the uncoated regions along the filaments, but caused only minor effects in the polyurethane coated regions. That follows from the observation that the tensile data for wet and dry conditions are very different for $C = 0$, but are essentially the same for $C = 1$.

The behavior may be modelled by saying that the wet uncoated interfacial shear strength $[(\tau_{uc})_{wet}]$ is altered considerably, whereas $(\tau_c)_{wet} \approx (\tau_c)_{dry}$. Then, considering the wet $C = 0$ and wet $C = 1$ results, we have from the rule of mixtures for the tensile strength of the unidirectional fiber samples,

$$\begin{aligned} 250 &= (1-v_f)\sigma_m + v_f\sigma_f \left\{ 1 - \frac{\psi}{2n} \right\} \\ \text{and} \quad 420 &= (1-v_f)\sigma_m + v_f\sigma_f \left\{ 1 - \frac{\psi}{2nT_{wet}} \right\} \end{aligned} \quad \left. \vphantom{\begin{aligned} 250 &= (1-v_f)\sigma_m + v_f\sigma_f \left\{ 1 - \frac{\psi}{2n} \right\} \\ 420 &= (1-v_f)\sigma_m + v_f\sigma_f \left\{ 1 - \frac{\psi}{2nT_{wet}} \right\} \right\} \text{ MN/m}^2$$

where now

$$T_{wet} = (\tau_c/\tau_{uc})_{wet}$$

The first equation of the pair may be solved for $(\psi/n)_{wet}$, which, using $v_f = 0.25$, $\sigma_m = 31 \text{ MN/m}^2$, $\sigma_f = 3.45 \text{ GN/m}^2$ gives $(\psi/n)_{wet} = 1.56$. (ψ/n) , it may be remembered, is equal to $\left\{ \frac{(l_{crit})_{uc}}{l_r} \div \frac{L}{l_r} \right\}$ where $(l_{crit})_{uc}$ is the critical transfer length of the uncoated fibers (in this case the wet critical transfer length), l_r is the repeat distance of the intermittent bond, and L is the finite size of the testpiece. Thus,

$$(\psi/n)_{wet} = \left\{ (l_{crit})_{uc} / L \right\}_{wet} = 1.56$$

Using this value in the second of the pair of equations, enables T_{wet} to be solved as $T_{wet} = 1.35$. That is, the interfacial shear strength of the polyurethane coated interfacial bond is stronger than the interfacial shear strength of the wet uncoated regions.

Since from previous dry investigations, T (meaning T_{dry} in the present con-

text) = 0.05 for the polyurethane coating, we have

$$(\tau_c/\tau_{uc})_{\text{wet}} = 1.35 \quad (\tau_c/\tau_{uc})_{\text{dry}} = 0.05$$

Whence if, as postulated, the coated regions are unaffected by water, τ_c may be eliminated between the relationships to give

$$(\tau_{uc})_{\text{dry}} = 27 (\tau_{uc})_{\text{wet}}$$

i.e., water has reduced the uncoated bond strength by a factor of 27.

It seems therefore that the opposing behaviors of the wet and dry data—the wet increasing in strength to the fully coated value of circa 420 MN/m^2 from the uncoated value of 250 MN/m^2 , but the dry falling from 650 MN/m^2 to about 450 MN/m^2 —may be explained in terms of different T's, with $T_{\text{dry}} < 1$ and $T_{\text{wet}} > 1$. T_{dry} is referenced to $(\tau_{uc})_{\text{dry}}$; T_{wet} is referenced to $(\tau_{uc})_{\text{wet}}$.

This may be checked out by noting that $d\sigma/dc$ from the rule of mixtures is proportional to

$$- [1 - c(1-T)]^2 (1-T)$$

so that $d\sigma/dc$ is positive for $T > 1$, and negative for $T < 1$.

4.2 Wet Critical Lengths

The critical lengths of the filaments, in their various wet conditions, may be determined as follows: the critical length of the uncoated dry specimens is, from previous studies, about 3.5 mm; this comes from $l_{\text{crit}} = \sigma_f d / 2\tau$ with $\sigma_f = 3.45 \text{ GN/m}^2$, $d = 140 \text{ }\mu\text{m}$ and $\tau = 69 \text{ MN/m}^2$. Since

$$(\tau_{uc})_{\text{wet}} = (1/27)(\tau_{uc})_{\text{dry}}$$

it follows that

$$((l_{\text{crit}})_{uc})_{\text{wet}} = 27 ((l_{\text{crit}})_{uc})_{\text{dry}}$$

i.e. about 95 mm. Also,

$$((l_{\text{crit}})_c)_{\text{wet}} \approx ((l_{\text{crit}})_c)_{\text{dry}}$$

because the tensile strengths of the fully coated samples, both wet and dry, are the same (circa $420 \sim 450 \text{ MN/m}^2$). The interfacial shear bond strengths of all fully-coated polyurethane samples (wet and dry) are the same therefore. From

previous studies T (in this context for 'dry' data) $= \tau_c / \tau_{uc} = 0.05$, so that the fully coated critical length is $((\ell_{crit})_c)_{dry} = \frac{3.5}{0.05} \text{ mm} = 70 \text{ mm}$. Or, equivalently in terms of T for wet data, i.e. τ_c as a proportion of the uncoated wet τ_{uc} , we have

$$(\tau_c / \tau_{uc})_{wet} = 1.35$$

so

$$((\ell_{crit})_c)_{wet} = \frac{((\ell_{crit})_{uc})_{wet}}{1.35} = \frac{95}{1.35} = 70 \text{ mm}, \text{ again}$$

The critical length of a general intermittently bonded specimen is given by

$$(\ell_{crit}) = (\ell_{crit})_{uc} / \{1 - C(1 - T)\}$$

which becomes for dry samples (earlier studies)

$$(\ell_{crit}) = \frac{3.5}{\{1 - 0.95C\}} \text{ mm}$$

because $T = 0.05$ referenced to dry uncoated interfaces, and

$$(\ell_{crit}) = \frac{95}{\{1 + 0.35C\}} \text{ mm}$$

because $T = 1.35$ referenced to wet uncoated interfaces. For $C = 1$, both expressions give the same ℓ_{crit} .

All the foregoing is predicated on using $\sigma = 420 \text{ MN/m}^2$ at $C = 1$ for the wet samples. In fact, our (limited) experiments give a higher value of σ for $C = 0.5$. In order to fair-in the results, calculations can be performed taking $\sigma = 420 \text{ MN/m}^2$ at $C = 0.5$ instead of at $C = 1$. This gives a better fit to the data when plotted in Figure 2(a). Then, $T = (\tau_c / \tau_{uc})_{wet} = 1.7$ instead of 1.35, whence

$$(\tau_{uc})_{dry} = 34 (\tau_{uc})_{wet}$$

and

$$(\ell_{crit}) = \frac{95}{\{1 + 0.7C\}} \text{ mm.}$$

This would make some difference to the critical lengths, the fully coated value

(C = 1) being some 56 mm instead of 70 mm.

We really require more data to get the σ vs. C relationship in greater detail, but the trends of what occurs in the wet condition are quite clear from the Figures and this analysis.

4.3 Effective Length of Tensile Specimen (L)

We have

$$\begin{aligned}\sigma &= (1-v_f)\sigma_m + v_f\sigma_f \left[1 - \frac{\psi}{2n\{1-C(1-T)\}} \right] \\ &= (1-v_f)\sigma_m + v_f\sigma_f \left[1 - \frac{(\ell_{crit})_{uc}}{2L\{1-C(1-T)\}} \right]\end{aligned}$$

for $(\ell_{crit})_{uc}$ wet or dry as appropriate. Now it was determined that $(\psi/n)_{wet} = (\ell_{crit})_{uc}/L_{wet} = 1.56$ from the C = 0 wet result. Thus, given

$$(\ell_{crit})_{uc,wet} = 95 \text{ mm}$$

so that L = 61 mm.

We note that the overall lengths of our tensile specimens were some 100 mm, with a shorter gauge length.

A comparable result follows from the dry data, because

$$(\psi/n)_{dry} = (\ell_{crit})_{uc}/L_{dry} = 0.05$$

Thus $L = \frac{3.5}{0.05} = 70 \text{ mm}$.

The dry tensile specimens (manufactured and tested much earlier) were roughly the same in size.

It must be pointed out that since the effective lengths of the wet tensile specimens (61 mm) is less than the critical length of the uncoated wet samples (95 mm), and only just about the critical length of the fully coated wet samples (70 mm), the 'simple' rule of mixtures, i.e.

$$\sigma = (1-v_f)\sigma_m + v_f\sigma_f$$

would markedly overestimate the observed strengths.

Longer tensile specimens would give higher results, but all would show the same changes with C as in Figures 2.

Because the thawed specimens displayed slightly lower strengths, but the same trends as the simply wet samples, it follows that the freezing/thawing cycle must have further weakened the interfacial shear strength, or caused physical separation of filament and matrix, or some physical separation of the layered testpieces (i.e. delamination).

4.4 Pull-Out Lengths

The changes in wet critical lengths are reflected in the filament pull-out lengths, both of the tensile specimens and also the toughness specimens, (to be discussed later). As is well-known, the longest pull-out length should be $(\ell_{crit})/4$, and the average pull-out length $(\ell_{crit})/2$, since the shortest pull-out length will be zero. Thus, the longest pull-out lengths should be $(95/2) = 48$ mm for the uncoated wet samples and $(70/2) = 35$ mm for the fully coated wet samples,—other samples with C between 0 and 1 taking intermediate values. Because $L < (\ell_{crit})$ for the wet samples, these full values for pull-out were not observed. Rather values of about 20 mm were seen for wet uncoated samples, with yet smaller values for samples that had C approaching unity.

A significant feature of the angle-ply laminates was that the 45° filaments broke off essentially in the main plane of fracture, and thus displayed virtually no pull-out lengths.

4.5 Toughness Analysis

4.5.1 Unidirectional and Angle-Ply Dry Results

It will be remembered that the total toughness is given by

$$R_{total} = R_{surfaces} + R_{redist} + R_{pull-out}$$

where $R_{surfaces}$ relates to debonding (mode II), R_{redist} relates to Piggott/Fitz-Randolph stress redistribution (or relaxation), and $R_{pull-out}$ relates to Cottrell/Kelly pull-out. An additional component, $R_{Cook/Gordon}$ must be added to the above if tensile debonding (mode I) takes place ahead of the running crack. The Cook/Gordon mechanism itself is a small toughness sink, but the associated additional

long debond lengths in the presence of Cook/Gordon debonding significantly increase the pull-out lengths and hence the total toughness. These aspects of toughness components were discussed in detail in the Final Report of this NASA Grant dated July 31st, 1974 and in a Journal of Materials Science paper*.

The differences in dry toughness levels of the intermittently bonded angle-ply laminates compared with the unidirectional specimens (described in Part I of this report, and also shown in Figures 3), may be explained partly in terms of loss of pull-out component of the 45° angle-ply (since, as described in section 4.4 the 45° angles break off in the plane of fracture with little or no pull-out). For example, both unidirectional and angle-ply uncoated specimens had $R_{\text{total}} \approx 40-50 \text{ kJ/m}^2$, whereas at $C = 1$, $R_{\text{total}} \approx 300 \text{ kJ/m}^2$ for the unidirectional specimens but only about 180 kJ/m^2 for the angle-ply testpieces. In a 5-layer testpiece, (forgetting the outside "crack path stabilizing" layers), two are 45° angle plies. Thus, in very rough terms, we should expect only 3/5 of the pull-out work that we would see in a 5-layer unidirectional specimen. At $C = 1$ in such a unidirectional laminate, $R_{\text{pull-out}} \approx 231 \text{ kJ/m}^2$ in polyurethane coated composites (see earlier report, or J. Mater. Sci. paper). Thus, in the angle-ply laminate, $R_{\text{pull-out}} \approx (3/5) 231 \approx 140 \text{ kJ/m}^2$. According to the analysis presented before, $R_{\text{surfaces}} \approx 18 \text{ kJ/m}^2$ and $R_{\text{redist}} \approx 100 \text{ kJ/m}^2$ for $C = 1$. We might expect therefore

$$R_{\text{total}} = 18 + 100 + (3/5)231 = 258 \text{ kJ/m}^2$$

It seems that the redistribution component is also being affected by the angle-ply geometry; were that component also reduced to (3/5) of the uniaxial value, R_{total} would be about 218 kJ/m^2 . [however, see comments on pp 14/15 about 'corrected' value]

4.5.2 Wet and Thawed Data

In regard to an analysis of the toughnesses of the wet and thawed samples (both unidirectional and angle ply), we are faced with the same difficulty as before, namely that interfacial toughnesses are important parameters, yet we have no direct

* J. Mater. Sci., 10 (1975), 819-832.

way of measuring them (in either mode I or mode II). Certainly, to infer interfacial toughnesses from interfacial shear strengths can lead to gross errors, as was demonstrated in the earlier studies by the fact that silicone vacuum grease and polyurethane varnish coatings seemed to produce comparable interfacial shear strengths, yet displayed quite different toughness behaviors. In the earlier work, it was loosely argued that if any relationship at all existed between interfacial τ 's and interfacial R 's, it probably was predicated on the interfacial stress intensity factors (K). That is, since the interfacial stress is proportional to some interfacial K , and if $K^2 = ER$ in the usual fashion, then $\tau \propto \sqrt{R}$.

Such a line of thought leads to the following: since

$$(\tau_{uc})_{wet} \approx (1/27) ((\tau_{uc})_{dry})$$

then

$$\begin{aligned} (R_{uc})_{wet} &\approx \frac{1}{27^2} ((R_{uc})_{dry}) \\ &\approx \frac{1}{730} ((R_{uc})_{dry}) \end{aligned}$$

The component of R_{surfaces} that concerns interfacial fractures is given by

$$v_f \cdot \frac{l_{\text{crit}}}{d} \cdot R_{\text{interface}}$$

For the wet uncoated case, $l_{\text{crit}} = 95 \text{ mm}$ (i.e. $27 \times (l_{\text{crit}})_{\text{dry}}$), $d = 140 \text{ } \mu\text{m}$ and $R_{\text{interface}} = \frac{1}{27^2} (R_{\text{interface}})_{\text{dry}}$ so that the toughness contribution from debonding the wet interface is

$$\frac{(0.25)(27 \times 3.5)10^{-3}}{140 \times 10^{-6}} \frac{1}{27^2} (R_{uc})_{\text{dry}} = 0.23 (R_{uc})_{\text{dry}}$$

It was assumed in earlier work that $(R_{uc})_{\text{dry}} \approx R_m \approx 2.6 \text{ kJ/m}^2$, so that in the wet uncoated case the debond surfaces contribution to R may only be about 0.6 kJ/m^2 .

At intermittent values of C , R_{surfaces} is given by

$$v_f \cdot \psi \lambda \left\{ \frac{1 - C(1 - T)}{1 - C(1 - T)} \right\} (R_{\text{if II}})_{uc}$$

Referencing quantities with respect to the wet uncoated case, $T = 1.35$ say,

$$p_{\text{I}} < T^2 = 1.8, \quad \psi \lambda = \frac{((l_{\text{crit}})_{uc})_{\text{wet}}}{d} = \frac{95 \times 10^{-3}}{140 \times 10^{-6}} = 680$$

so for $v_f = 0.25$,

$$R_{\text{surfaces}} = (0.25)(680) \left\{ \frac{1 + 0.8C}{1 + 0.35C} \right\} \frac{2.6}{27^2} \text{ kJ/m}^2$$

which for $C = 1$, becomes 0.8 kJ/m^2 . This is really quite small compared with the total measured values. In fact, the contribution is even less because the half-height of the toughness specimen (38 mm) is less than half the critical lengths, so that full 'potential' debonding is never attained.

It seems therefore that of all the surfaces components, probably the most significant is that of the matrix itself, at $(1 - v_f) R_m \approx 2 \text{ kJ/m}^2$.

Regarding the Piggott/Fitz-Randolph redistribution contribution, we have

$$R_{\text{redist}} = \frac{v_f \sigma_f^2 \psi \lambda d}{6 E_f [1 - C(1 - T)]}$$

for boron/epoxy systems, where the filament fracture strain is less than the matrix fracture strain. Referencing quantities to the wet uncoated condition, we have

$$(\psi \lambda d) = ((l_{\text{crit}})_{uc})_{\text{wet}} = 95 \times 10^{-3} \text{ m}, \quad T = 1.35, \quad \sigma_f = 3.45 \text{ GN/m}^2, \quad E_f = 380 \text{ GN/m}^2.$$

Thus

$$R_{\text{redist}} = \frac{(0.25)(3.45 \times 10^9)^2 95 \times 10^{-3}}{6 \times 380 \times 10^9 (1 + 0.35C)}$$

$$= \frac{30.7}{1 + 0.35C} \text{ kJ/m}^2$$

which for $C = 0$ is 30.7 kJ/m^2 and for $C = 1$ is 23 kJ/m^2 .

Since the critical lengths of the filaments in their various wet intermittently bonded conditions are all greater than the specimen height, R_{redist} should be reduced below the foregoing values. Given that random filament fracture, on average, should occur at $\pm \ell_{\text{crit}}/4$ above or below the main crack plane, it is clear that broken filaments have only $\{38 - \ell_{\text{crit}}/4\} \text{ mm}$ and $\{38 + \ell_{\text{crit}}/4\} \text{ mm}$ on which irreversible interfacial slip can take place. Given the fact that Piggott/Fitz-Randolph stress relaxation is predicated on irreversible slip over interfacial lengths ($\ell_{\text{crit}}/2$) on either side of the filament fracture, it seems that one side of a broken filament can only produce a share $\left\{ \frac{(38 - \ell_{\text{crit}}/4)}{\ell_{\text{crit}}/2} \right\}$ towards $1/2 R_{\text{redist}}$, and the other side $\left\{ \frac{38 + \ell_{\text{crit}}/4}{\ell_{\text{crit}}/2} \right\}$ towards $1/2 R_{\text{redist}}$. Thus the corrected R_{redist} is

$$\left\{ \frac{38 - \ell_{\text{crit}}/4}{\ell_{\text{crit}}/2} \right\} \frac{1}{2} R_{\text{redist}} + \left\{ \frac{38 + \ell_{\text{crit}}/4}{\ell_{\text{crit}}/2} \right\} \frac{1}{2} R_{\text{redist}}$$

for ℓ_{crit} in mm. This becomes

$$\left\{ \frac{76}{95} \right\} (1 + 0.35C) R_{\text{redist}}$$

because ℓ_{crit} at any C is $\{(\ell_{\text{crit}})_{\text{uc}}^{\text{wet}} \div [1 - C(1 - T)]\}$.

Now

$$R_{\text{redist}} = \frac{30.7}{(1 + 0.35C)} \text{ kJ/m}^2$$

which gives, for the corrected result, the constant value of circa 25 kJ/m^2 —independent of C , for the particular size specimens used.

Pull-out toughness is given by

$$R_{\text{pull-out}} = \nu_f \tau' \lambda h \left[\frac{\psi}{\{1 - C(1 - T)\}} + 2C \right]$$

where h is the crack opening at the propagated crack length used in the Gurney

segmental area method (say 0.5 mm), and τ' is the interfacial frictional stress during pull-out (values of which have been given in section 3, at various C). Thus for the wet samples

$$R_{\text{pull-out}} = (0.25) \tau' \left\{ \frac{25.4 \times 10^{-3}}{140 \times 10^{-6}} \right\} 0.5 \times 10^{-3} \left[\frac{95 \times 10^{-3}}{25.4 \times 10^{-3} (1 + 0.35C)} + 2C \right] \\ = \tau' \left[\frac{8.5 \times 10^{-5}}{1 + 0.35C} + 4.5 \times 10^{-5} C \right] \text{ kJ/m}^2$$

For $C = 0$, $\tau' = 0.5 \text{ MN/m}^2$, so

$$R_{\text{pull-out}} = 42.5 \text{ kJ/m}^2$$

For $C = 1$, $\tau' = 2.75 \text{ MN/m}^2$, so

$$R_{\text{pull-out}} = (173 + 124) = 297 \text{ kJ/m}^2$$

or, using $\tau' = 2 \text{ MN/m}^2$ (earlier work),

$$R_{\text{pull-out}} = (126 + 90) = 216 \text{ kJ/m}^2$$

Again, because the size of the specimens is comparable to, or less than, the critical lengths, and because the pull-out lengths with Cook/Gordon debonding are larger than the half-heights of the specimens, the full potential contribution to toughness from pull-out may not be achieved. The J. Mater. Sci. paper showed that

$$R_{\text{pull-out}} \approx 4 \nu_f \tau' D h / d$$

where D on average was $\ell_{\text{crit}}/4$ in the absence of Cook/Gordon debonding, or was

$\{\ell_{\text{crit}}/4 + C \ell_r/2\}$ on average in its presence, where ℓ_r is the repeat distance.

Only when $D < 38 \text{ mm}$ (the half height of the toughness specimen) would full pull-out be attained, otherwise it would be merely the frictional work over the finite 38 mm.

For wet uncoated samples $\ell_{\text{crit}} = 95 \text{ mm}$, so with $\ell_r = 25 \text{ mm}$, $D = 95/4 + 25/2 = 36 \text{ mm}$,

or with $\ell_r = 51 \text{ mm}$, $D = 49 \text{ mm}$. For wet coated samples, $\ell_{\text{crit}} = 70 \text{ mm}$, so $D = 70/4 + 25/2 = 30 \text{ mm}$ with $\ell_r = 25 \text{ mm}$, or $D = 43 \text{ mm}$.

Note that these calculations are based on averages, i.e. that some filaments break with zero pull out (in the plane of gross fracture) and others break with the

longest possible pull-out of $\ell_{crit}/2$ (all subsequently augmented by Cook/Gordon pull-out lengths). Therefore, even though with $\ell_r = 25$ mm, the average D is less than 38 mm, the size of specimen does not allow the longest pull-out lengths ($95/2 + 25 = 73$ mm for $C = 0$, and $70/2 + 25 = 60$ mm for $C = 1$) to occur. This must skew the average distribution of pull-out lengths and reduce the potential toughness contribution. Assuming a Gaussian normal distribution between 0 mm pull-out lengths and 73 mm pull-out lengths for $C = 0$, the reduction is the (fractional) area under the distribution curve between zero pull-out and 38 mm pull-out in terms of the total area between zero and 73 mm pull-out. This is roughly 0.5, so that for $C = 0$, the effective pull-out contribution may be

$$R_{pull-out} = (0.5)(42.5) = 22 \text{ kJ/m}^2$$

Also, for $C = 1$, the proportional correction would be the area under the normal distribution curve between zero pull-out and 38 mm pull-out, divided by the total area between zero pull-out and 60 mm pull-out. From tables, that is about 0.67, so that at $C = 1$, the effective pull-out contribution may be

$$\begin{aligned} R_{pull-out} &= (0.67)(216 \text{ or } 297) \\ &\approx (144 \text{ or } 198) \text{ kJ/m}^2 \end{aligned}$$

Thus, for the total toughness of unidirectional wet composites we might expect, using the corrected values, for $C = 0$

$$R_{total} \approx 2 + 25 + 22 \approx 49 \text{ kJ/m}^2$$

and for $C = 1$,

$$R_{total} \approx 2 + 25 + (144 \text{ or } 198) = 171 \text{ or } 225 \text{ kJ/m}^2$$

For the total toughness of angle ply composites, arguing that the two 45° layers give no pull-out contribution, we have for $C = 0$

$$R_{total} \approx 2 + 25 + (3/5)22 \approx 40 \text{ kJ/m}^2$$

and for $C = 1$,

$$R_{\text{total}} = 2 + 25 + (3/5)(144 \text{ or } 198) = 114 \text{ or } 147 \text{ kJ/m}^2$$

Lines following these relations have been superimposed on Figures 3.

Again, reverting to the differences between unidirectional and angle-ply dry results (section 4.5.1), it is likely that the pull-out value should have been corrected along the foregoing lines to recognise the fact that the specimen size was comparable to the critical length. That is for $C = 1$, the unidirectional 5-layer 231 kJ/m^2 pull-out work should be reduced to about $(0.67) (231) = 55 \text{ kJ/m}^2$ before multiply by the "active" 3 layers in 5. Then

$$R_{\text{pull-out}} = (3/5)(155) = 93 \text{ kJ/m}^2$$

which gives for the total toughness of the fully coated dry angle-ply composite (see p. 10)

$$R_{\text{total}} = 18 + 100 + 93$$

This agrees much better with the data in Figure 3(b), without amending the redistribution component.

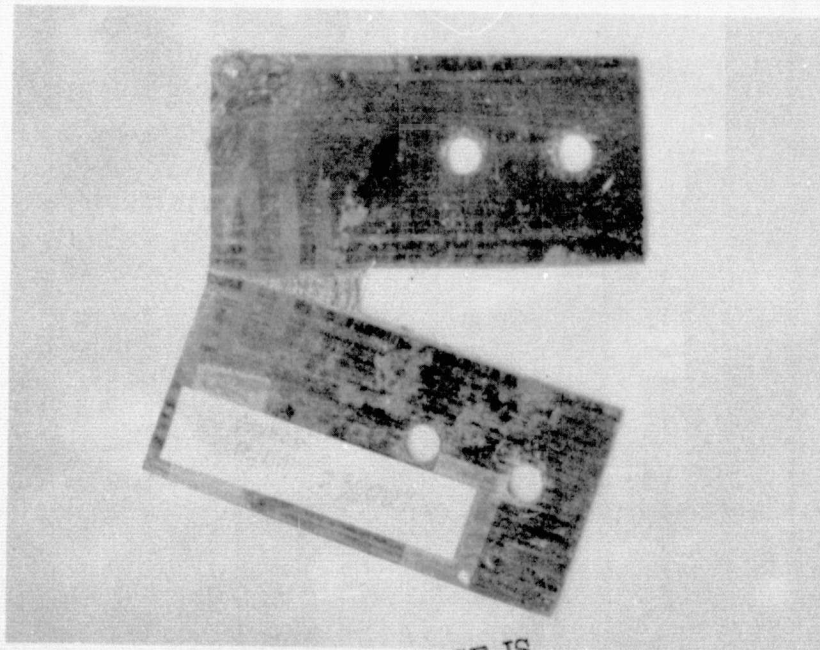
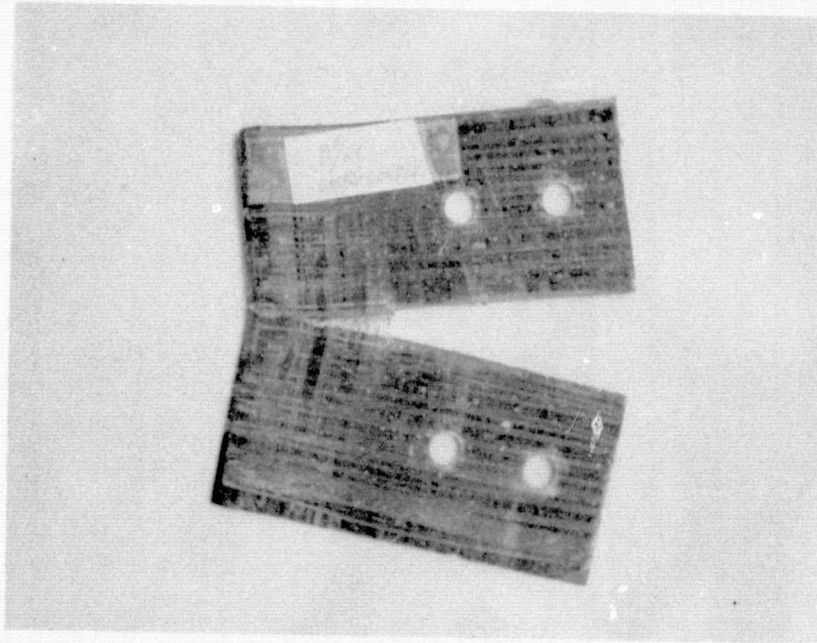
5. Conclusions

Although the old specimen sizes are not large enough to display the full potential strength and toughness behavior of the wet (and thawed) boron/epoxy unidirectional and angle-ply samples, it is clear that the extreme consequences of soaking with water are to

- (i) reduce markedly the tensile strength of the uncoated composites
- (ii) make hardly any difference to the composites fully coated with polyurethane varnish
- (iii) make hardly any difference to the uncoated toughnesses
- (iv) cause only a comparatively small reduction in the toughnesses of the fully coated samples.

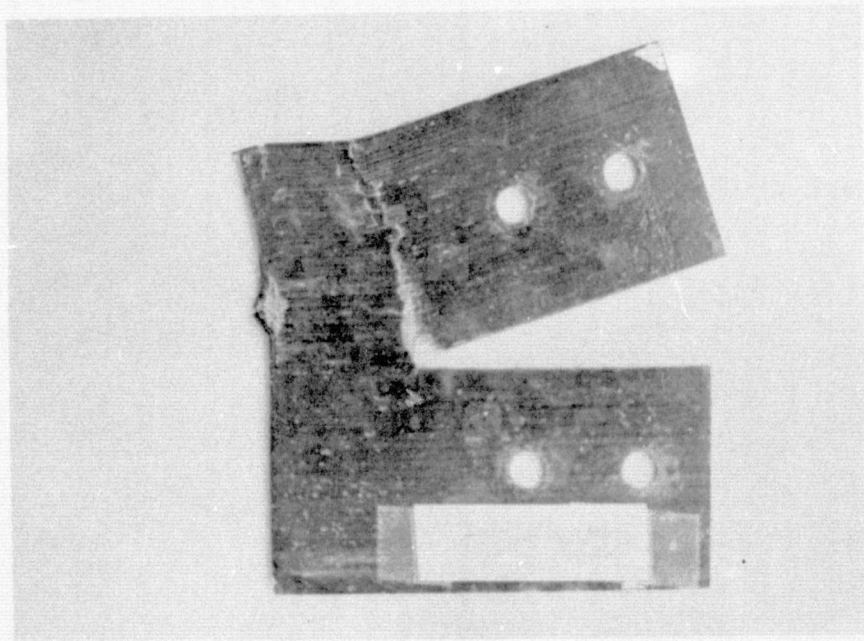
The effect of one freezing/thawing cycle is to reduce all strength and toughness values by a small amount. The effects of repeated freezing/thawing cycles is not known.

It seems very important, given that respectable wet toughnesses are still attainable, that the tensile strengths of fully coated polyurethane varnish specimens are the same whether wet or dry. Although there is a loss relative to the dry uncoated strength, that value ($600-650 \text{ MN/m}^2$) is not of much use if, upon getting soaked, it is reduced to only some 250 MN/m^2 .

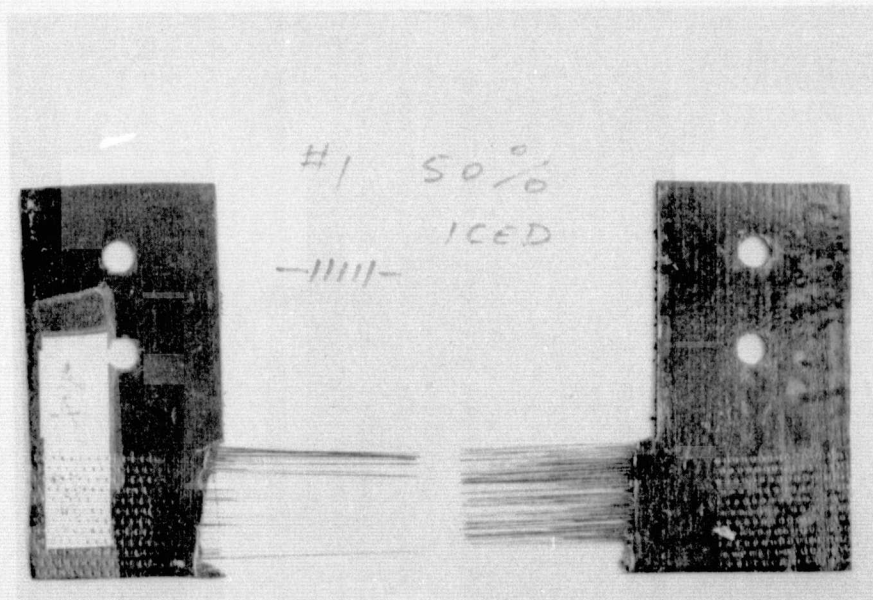


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Water Soaked Toughness Testpieces (Top) C = 0, (Bottom) C = 1.

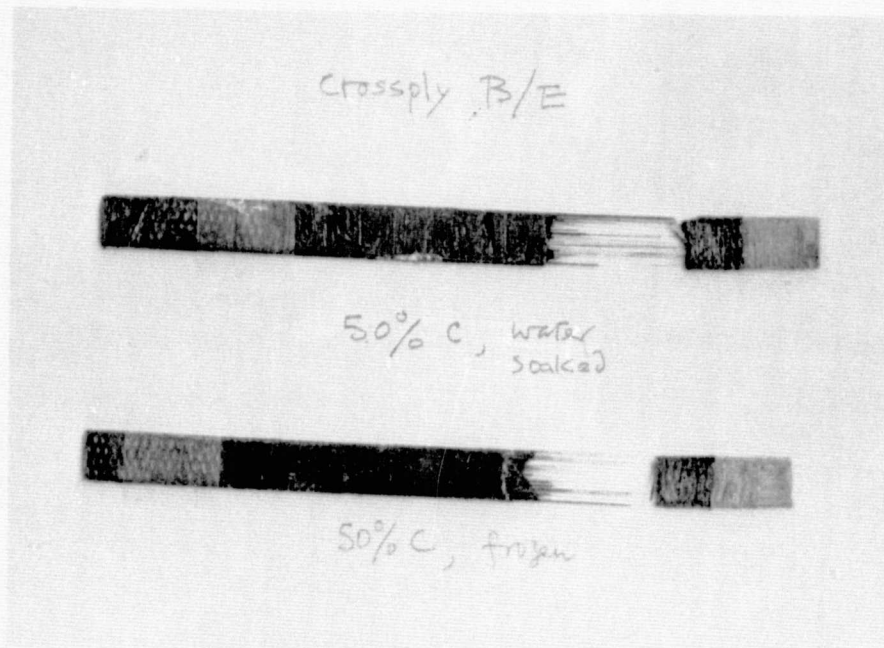


Water soaked $C = 0.5$ testpiece displaying side cracking.



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Experiment to determine average interfacial τ' during pull-out
(specimens regripped in line with remaining crack ligament).



Water soaked (top) and frozen/thawed (bottom) angle-ply tensile specimens ($C = 0.5$). Appreciable pull-out of longitudinal filaments, but hardly any for 45° angle-plyies.

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Legends for Figures

1. Water absorption isotherms.
2. (a) Tensile strengths of wet and thawed unidirectional filament testpieces.
(b) Tensile strengths of wet and thawed angle-ply testpieces.
3. (a) Fracture toughness of wet and thawed unidirectional filament testpieces.
(b) Fracture toughness of wet and thawed angle-ply testpieces.

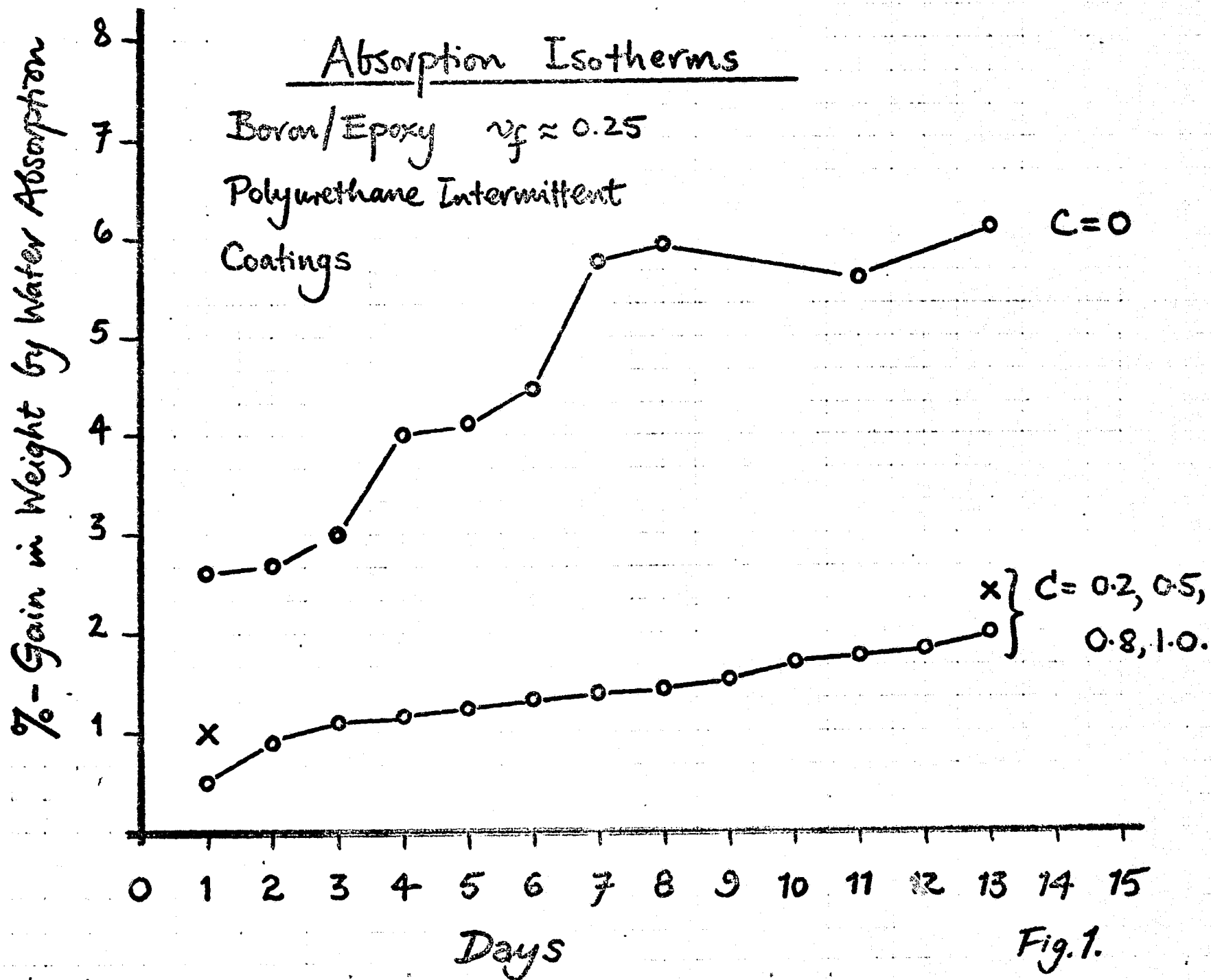


Fig.1.

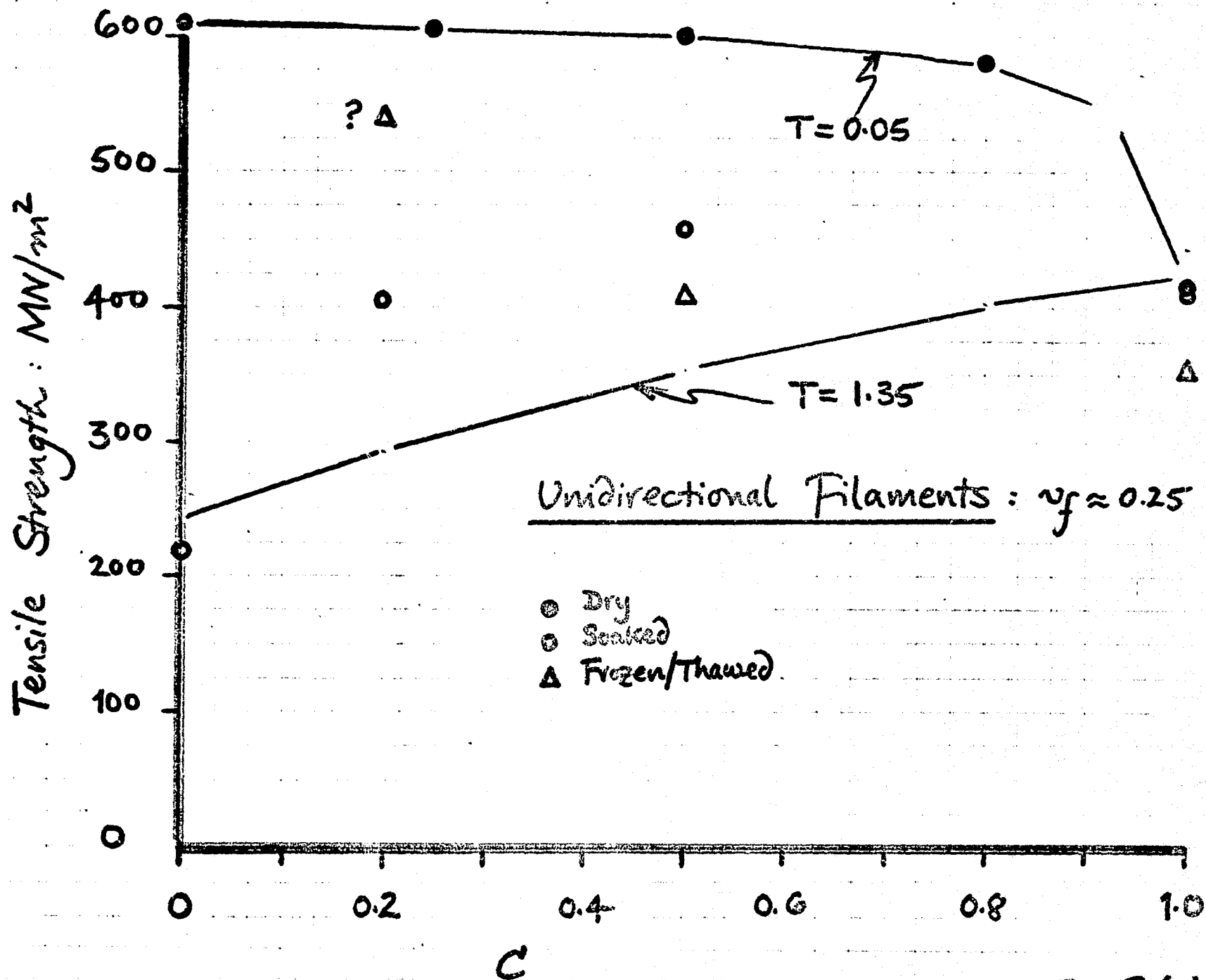


Fig. 2(a)

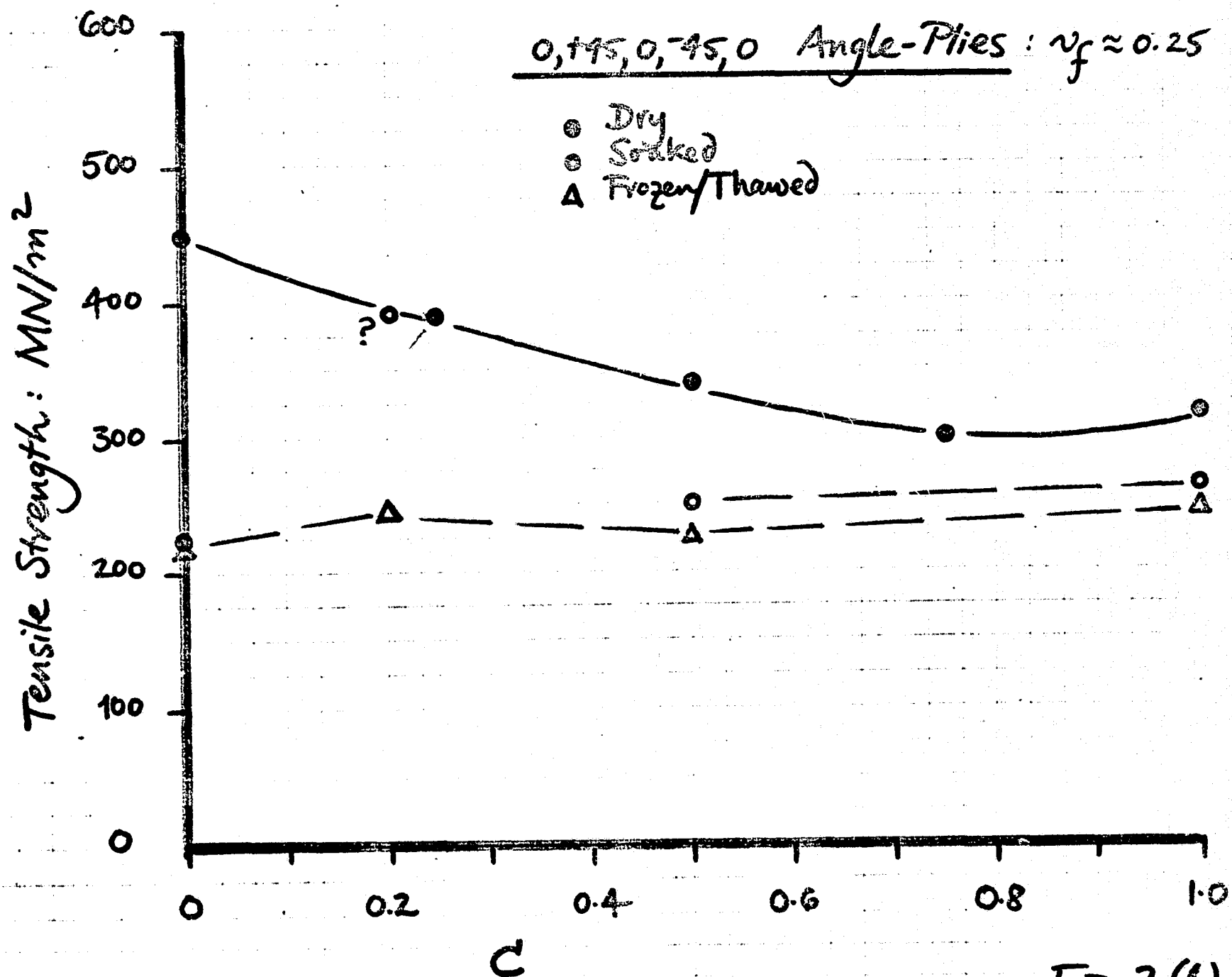


Fig. 2 (b)

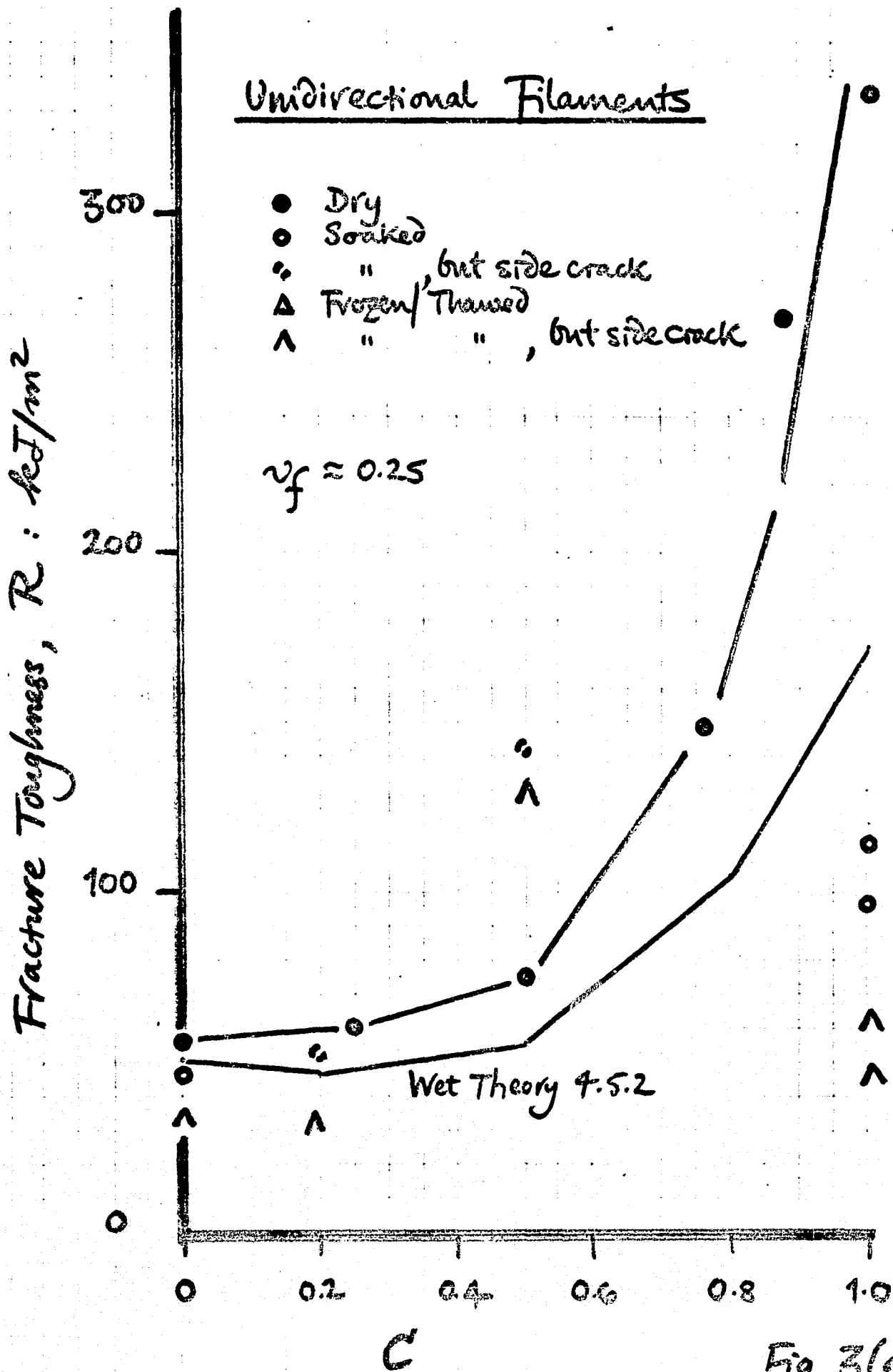


Fig. 3(a)

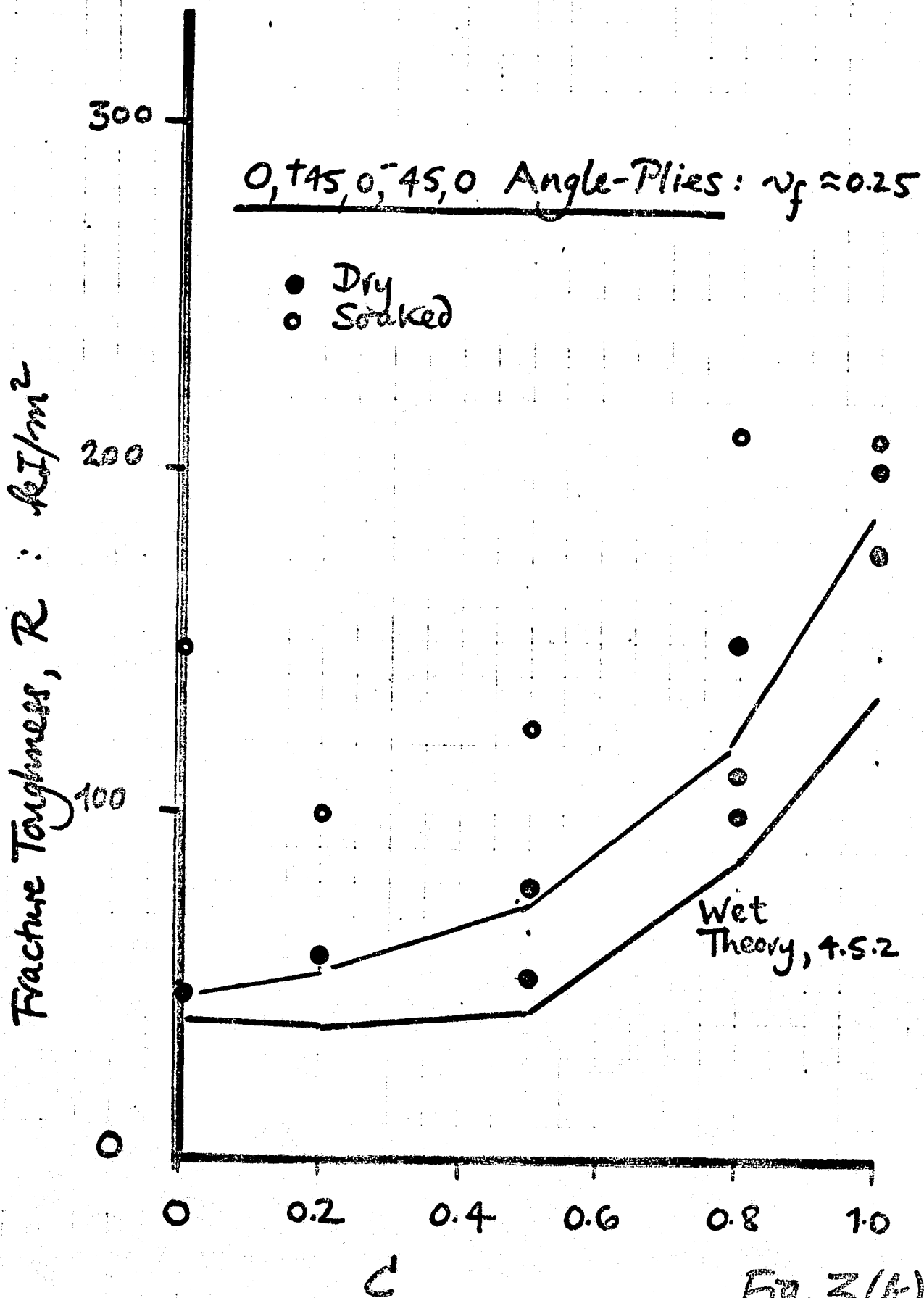


Fig. 3(A)

Part III

New Tape Making Apparatus

Introduction

Various views of the rebuilt apparatus are shown in the photographs. The principal difference from the apparatus described in the report of July 31, 1974, is the width of the intermittently bonded "pre-preg" tape, i.e. 230 mm (9 inch) as opposed to 77 mm (3 inch). In order to keep tape manufacture time roughly the same as before, three lots of filaments are wound at once, so that the new apparatus really makes three, 3-inch, tapes alongside one another. This arrangement allowed the same traversing carriage to be used as before, with two additional outrigger arms containing guides for the two additional reels being fed on to the wider drum.

The following description of tape manufacture was prepared by Mr. R. Cisler as a project in the Winter Semester, Academic Year 1974/75.

OPERATION OF THE
COMPOSITE TAPE APPARATUS
AND SPECIMEN CONSTRUCTION

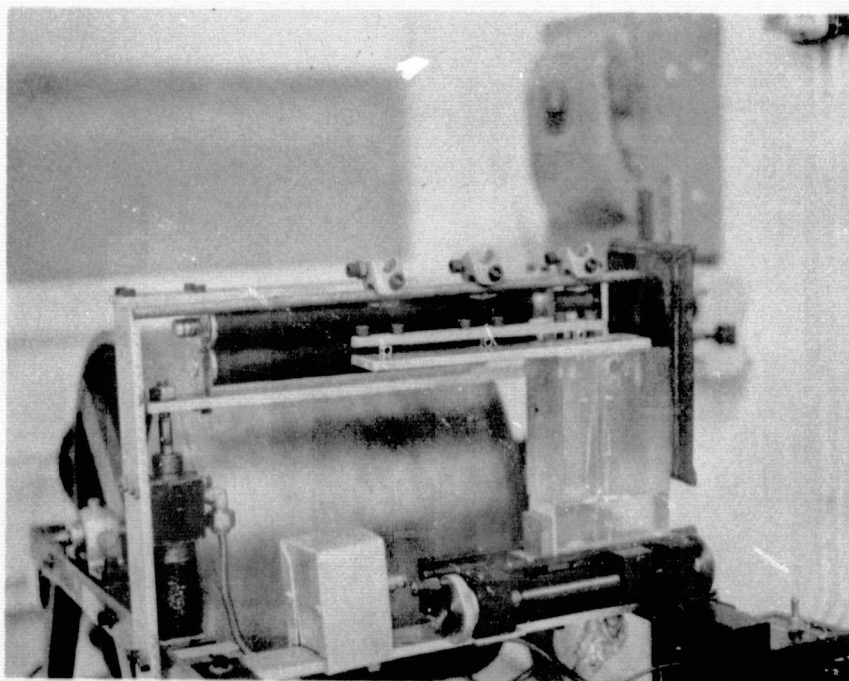
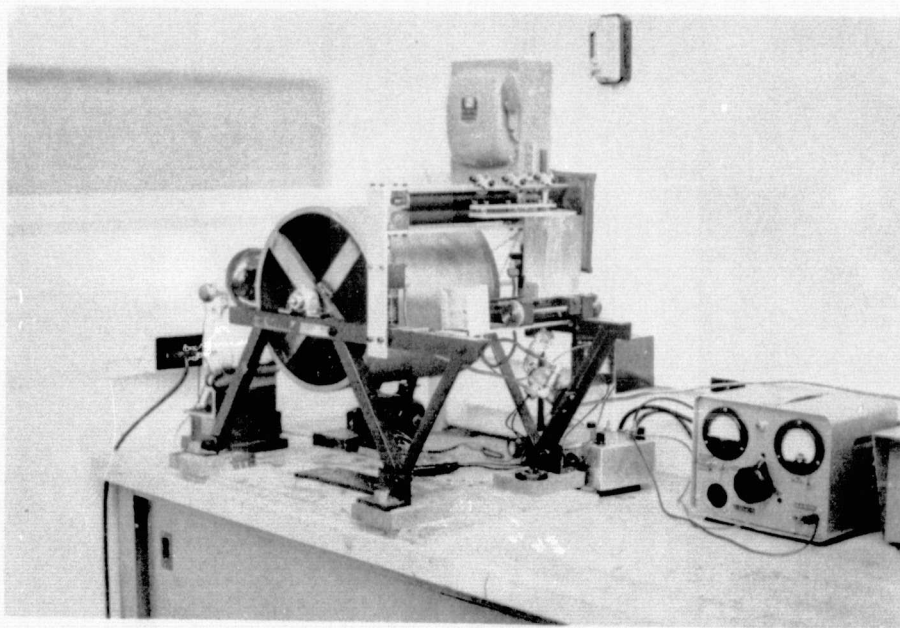
ME 600

Robert Cisler

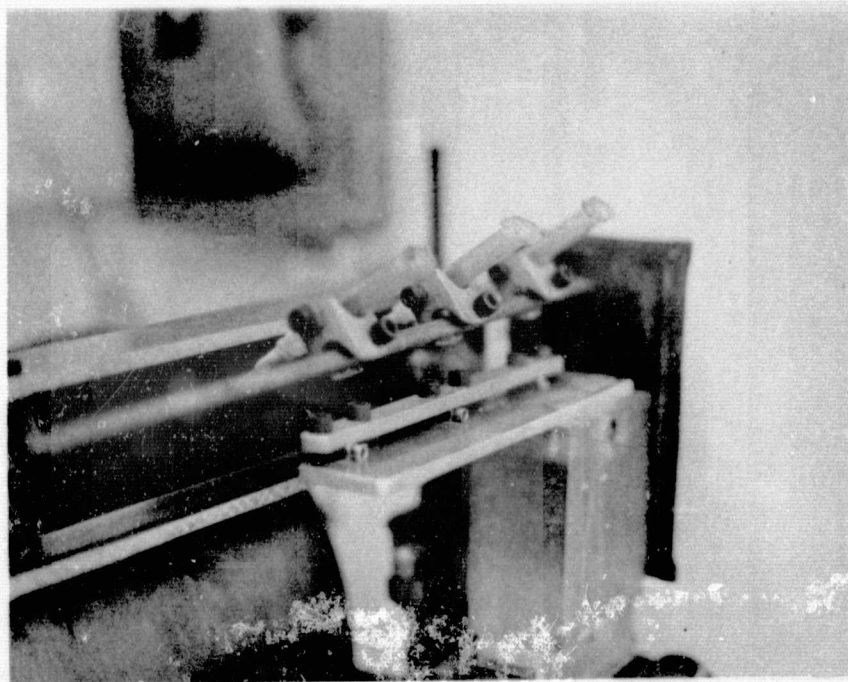
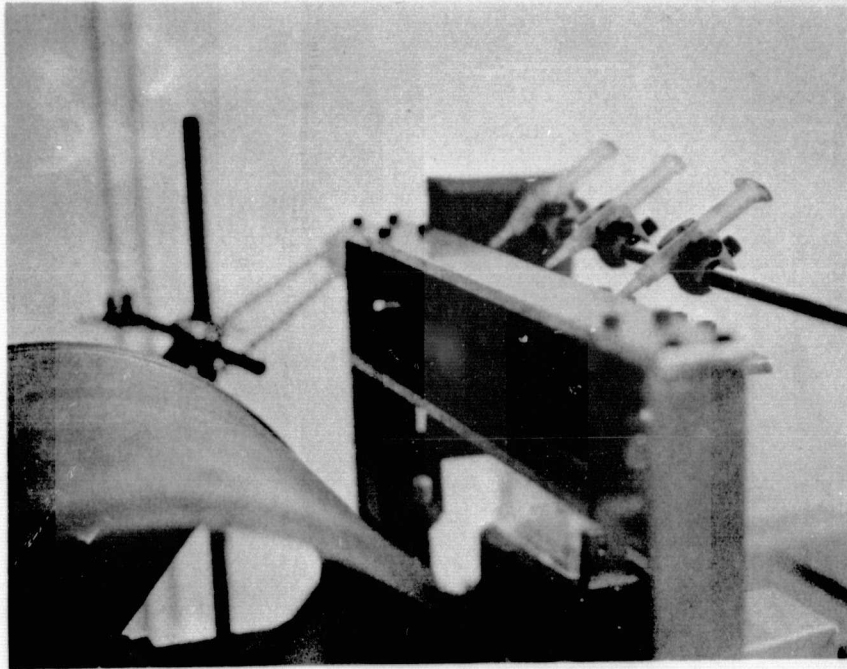
May 30, 1975

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Coating control settings	12
Specifications for fiber directions	13

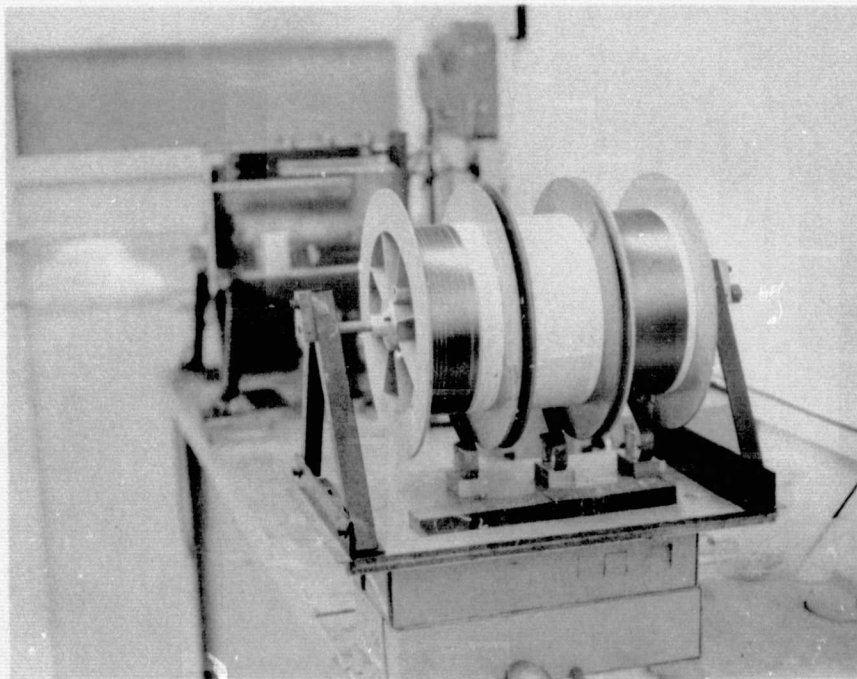
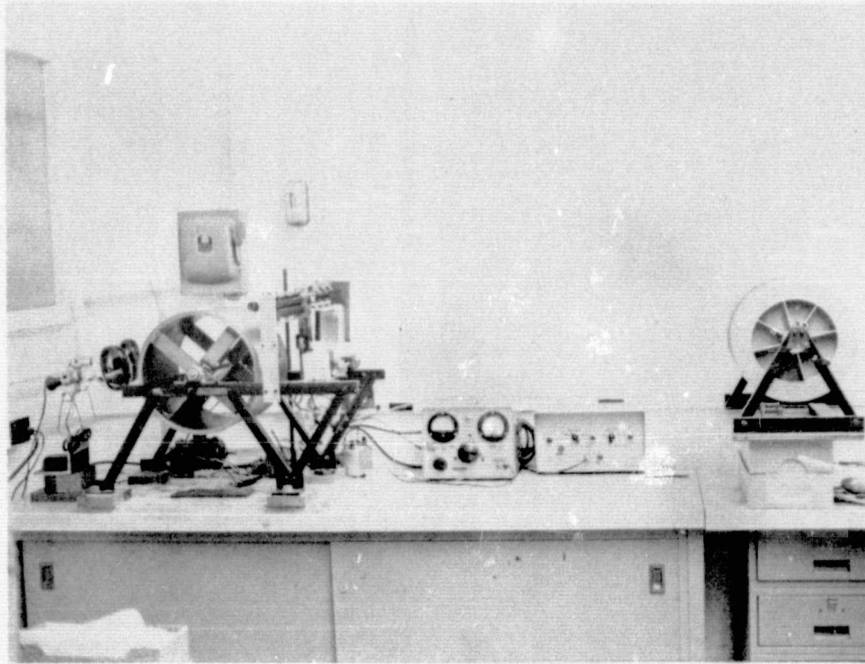


General Views of Tape Making Apparatus.



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General Views of Tape Making Apparatus.



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General Views of Tape Making Apparatus.

Operation of the Composite Tape Apparatus

The purpose of this paper is to provide a step by step procedure for both the operation of the composite tape-making apparatus and the construction of various composite specimens. The entire operation can be divided into three categories: preliminary work, composite tape construction, and specimen preparation.

Preliminary Work

The preliminary work consists of preparing the drum for winding and mixing and storing the epoxy for use later. Before each tape is made, the amount of fibers on each fiber reel must be checked. For U of M tapes ($V_f = .25$), two complete fiber layers on each reel are required while the N.A.S.A. tapes require 3 complete layers on each reel. If replacement of one of the fiber reels is necessary, the reel bearing arrangement is given in Figure 1. The reel bearings, coating roller shafts and bearings, and drum bearings must be kept well lubricated for the construction of each tape.

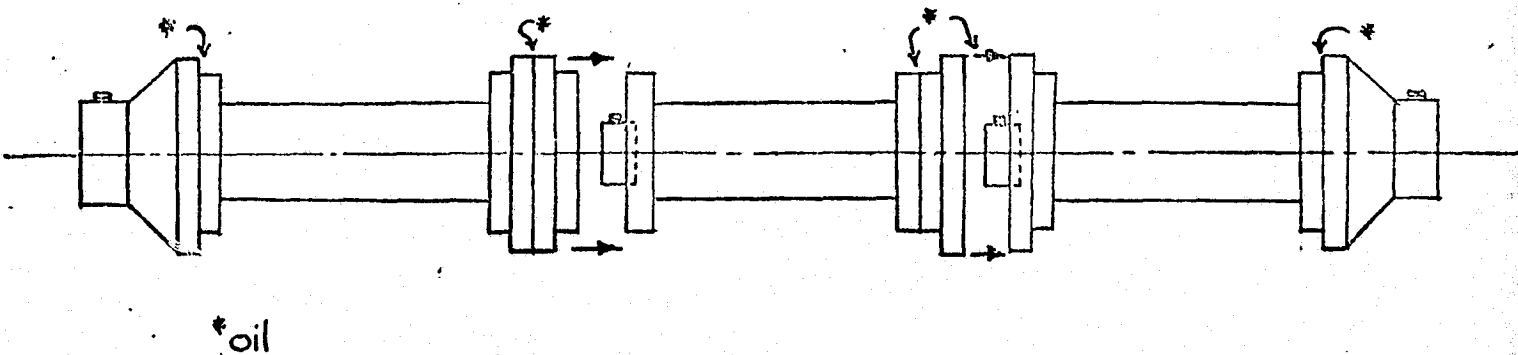


Figure 1.

The drum must now be cleaned of dried epoxy by scraping a razor blade over the surface while slowly rotating the drum. Now, wipe the surface clean with acetone. After stopping the drum with the three small holes in the position shown in Figure 2, spray a small amount of oven coating over the section

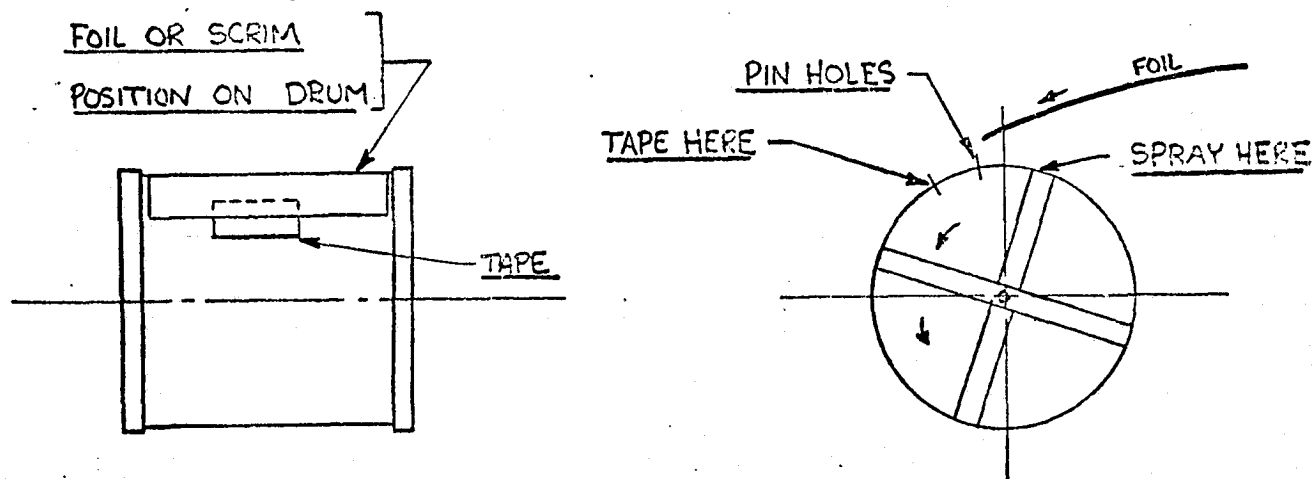


Figure 2.

of the drum where the finished tape is to be cut. This prevents the aluminum from sticking to the drum after the tape has been cut. Next place a piece of double stick tape about 1-2 inches from the 3 small holes in the center of the drum as shown in Figure 2. Then cut a piece of aluminum foil 9 1/2 inches x 1 meter and a piece of nylon scrim cloth 9 3/8 x 1 meter. A large number of these pieces may be cut and stored for use for subsequent tape construction. Center the aluminum foil on the drum, shiny side outward, and stick one end of the foil to half of the double-stick tape as shown in Figure 2. Rotate the drum slowly, keeping the foil centered, until the surface of the drum is covered. Then pull the center of the loose end and stick it to the other half of the double-stick tape. Tape the corners of the foil down with small pieces of double sided tape. Place another piece of double stick tape approximately 1 inch from the previous piece in the center and repeat the previous procedure

for the nylon scrim cloth. It may be necessary to use larger pieces of cellophane tape to hold the scrim joint together until the epoxy is applied.

The preparation of the epoxy for storage in the freezer is done by first preheating the vacuum oven to 60°C at a vacuum of 10-15 mm Hg. Set the temperature control at about 1.75 and monitor the temperature with a thermometer laying diagonally on the shelf. While the oven is heating, mix 80 grams of Shell-828 Resin with, 20 grams of the "Z" hardener in a large beaker and stir with a metal spatula. Larger quantities can also be made. Place this beaker into the oven and heat at 60°C \pm 5°C and 10-15 mm Hg vacuum for 15 minutes. Due to the instability in the temperature control it is often convenient to turn the oven off at this point to maintain a constant 60°C temperature in the oven. After the 15 minutes, increase the vacuum to 25-30 mm Hg and hold for another 5 minutes. Then remove the beaker from the oven and pour in 25 gram amounts in smaller beakers. Record the amount and date on the beaker and store in the freezer until needed. If a tape is to be made immediately this fresh epoxy may be used without freezer storage.

Coating the drum with epoxy first involves heating the slowly rotating drum with a heat lamp approximately 4" - 5" away from the rotating surface. Heating takes approximately 20 minutes and is necessary to keep the epoxy from cooling quickly through the steel surface, making spreading of the epoxy over the surface virtually impossible. About mid-way into the heating of the surface, remove the epoxy from the freezer and place it under a heat lamp to thaw. Keep checking the epoxy and remove the beaker from the direct heat when it is fluid enough to spread. If the epoxy is left too long under the heat it will begin to harden. (Note: If the epoxy is heated and then refrozen for some reason it will tend to harden extremely fast under slight amounts of re-heating.)

Suggested Amounts of Epoxy Coverage:

0° - 45° - 90° tapes : 50 grams epoxy

0° - 90° tapes : 45 grams. (50 grams can also be used to simplify the storage situation).

Once the epoxy has become sufficiently fluid to spread, turn off the heat lamps and with the drum still rotating, spread the epoxy over the drum roughly with a metal spatula. It is suggested that the epoxy first be placed on the scrim tape joint so that the reinforcing tape may be removed. Smooth the epoxy evenly over the entire scrim surface with the shielded side of a single edge razor blade. Make sure that the sharp side of the razor is taped before you attempt to use it.

To begin winding, stop the drum so that the three small holes on the drum surface are facing upward. Turn the air pressure to 40-50 psig. and set the rate control on "X10" to open the coating rollers. Locate the free end of the fiber on one of the spools and string it over to the drum leaving 8-10 inches excess as shown in Figure 3. Keep the fiber taunt until it is taped at the

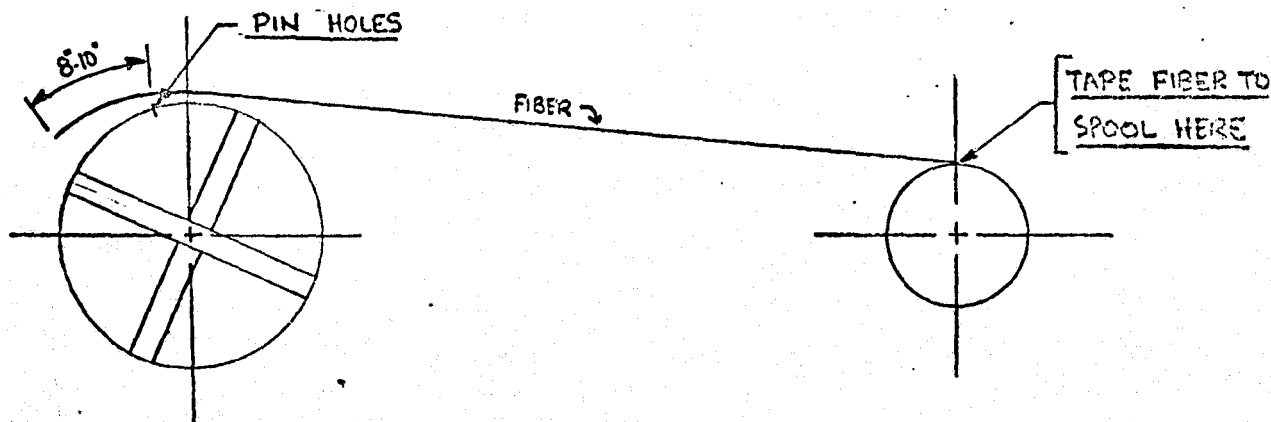


Figure 3.

spool to prevent unraveling. Repeat for the other spools. Thread the fibers through their respective nozzles and pull each between the open coating rollers. Now insert the three spring anchor pins through the holes in the drum and thread each fiber through the small hole at the end of the pin. A pair of tweezers may be needed to help break each pin through the foil and scrim layer. Pull the extra fiber straight back and tape it to the epoxy coated surface. Lightly tap this extra fiber along its length to assure that it is set in the epoxy. Remove the tape from the fibers at the spools, set the D.C. voltage rheostat to "0", and set the traverse on "off." Slowly increase the D.C. voltage until the drum begins to turn. Pushing the drum by hand aids in smooth starting which is important because jerky starts result in breakage of the brittle fibers at the anchor pins. Once the drum has started, continue to turn it at a very slow and smooth rate for 1 revolution. Then stop the drum and remove the tape holding the extra fiber (8"-10").

At this point the coating material is applied. Make sure the hypodermic needles are unclogged and then fill them with the polyurethane varnish (REZ). Adjust the needles in the holders as in Figure 4. The settings for the various

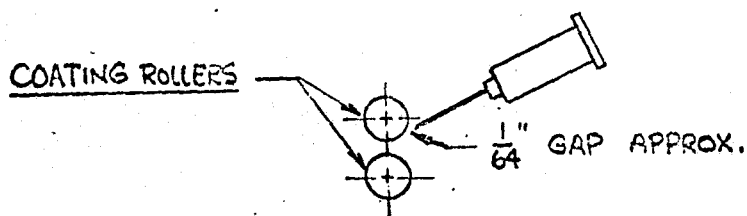


Figure 4.

coating percentages at various repeat distances is given at the end of this paper. The repeat distance used on both the U of M tapes ($V_f = .20 - .25$) and the N.A.S.A. tape ($V_f = .45 - .50$) is one inch. Set the coating controls to the desired setting. Reset the rate knob to the "X10" setting to open the rollers.

Now increase the D.C. voltage until the desired speed is reached. The

speeds for the frequently used volume fractions are listed below:

V_f	D.C. Volts
.20 - .25	60V (U of M)
.45 - .50	145V (N.A.S.A.)

Quickly turn the traverse switch to the "down" position and change the rate control back to the "X100" setting. For the first few minutes make sure the fibers wind correctly by manually steering them into the right lay direction (i.e. no overlap). This winding should be constantly monitored. Also make sure the coating needles are kept unclogged and filled with varnish.

When the winding is finished the traverse limit switch will automatically stop the traverse carriage motion. Stop the drum in approximately the position shown in Figure 5 and remove and clean the hypodermic needles. Place two pieces of cellophane tape across the full length of the composite tape as

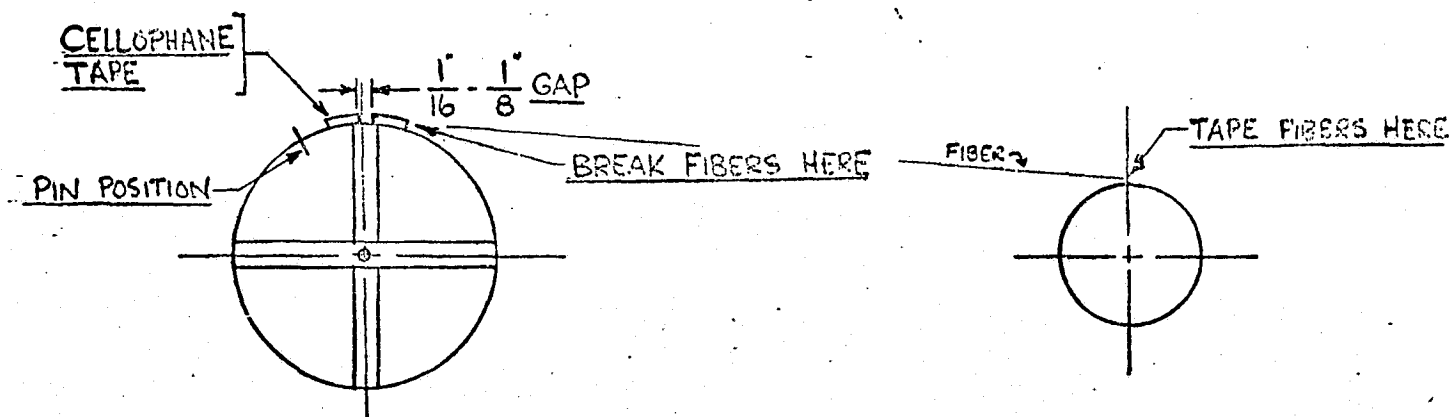


Figure 5.

shown in Figure 5 to prevent unraveling of the fiber upon cutting. Again tape the fibers at the spool and then break the fibers at the position shown in Figure 5. Rewind the fibers on their spools and tape each fiber end in an accessible place. Then cover the spools with the foam rubber padding. Remove

the spring anchor pins and place them in a tray of acetone to make sure the small holes are kept unplugged. Use a straight edge to guide glass cutter between the pieces of cellophane tape. Once the composite tape is cut, free the right side of tape (aluminum, scrim, and fibers) and while holding this side rotate the drum slowly until the entire tape peels free from the drum. Fold this tape as shown in Figure 6 and tape and label the tape before replacing



Figure 6.

it into the freezer. Disconnect the drive gear on the tranverse carriage and rewind the carriage by hand. Reconnect the drive gear and clean the entire drum and apparatus for the next tape-making operation. All components including the coating rollers and drum surface may be cleaned with acetone.

The peeling and cutting operation is the most tedious task in the entire tape making process. Many different procedures were tried throughout the semester but the one that follows seemed to be the most convenient.

The initial step involves unrolling the sheet of tin the full length of the work table and placing the tape from the freezer, fibers down, onto the tin sheet. If the tape sticks excessively to the tin use oven spray on the tin before placing the tape on it. Peel the aluminum foil off the tape (leave the scrim cloth attached to the fibers) until the epoxy becomes too tacky and unmanageable. At this point place the entire tape back into the freezer for a few minutes to allow the epoxy to reset. Then remove the tape and resume peeling the aluminum. Once the foil is completely removed, the tape is ready to be cut into pieces for the specimen.

At this point it can not be emphasized enough that THE ALUMINUM FOIL MUST BE PEELED OFF FIRST BEFORE ANY CUTTING IS ATTEMPTED! This particular point was violated in the beginning and it lead to many extra hours of tedious picking of aluminum foil from the backs of the cut pieces.

If you are not ready for cutting, store the peeled tape with a piece of wax paper placed "loosely on the back," similar to that pictured in Figure 6. When you are ready to cut the tape, remove the wax paper backing and place the tape fiber side down on the tin sheet. Again if excessive sticking occurs it may be necessary to use oven coating on the tin sheet before placing the composite tape down on it. Mark each tape according to one of the patterns provided in the lab note book. A flair pen works adequately for constructing the lines and a 45° triangle works well for the angle required. Use a straight edge and glass cutter to cut along the pattern lines. If specimens are to be molded a short time after cutting, the cut sections can be merely stacked in the freezer until they are used. For longer storage periods each piece should be placed loosely on a wax paper backing and clearly labeled.

Specimen Molding

Three types of specimens are molded : the 3x3 inch edge crack; the 3/4x4 1/2 inch tensile; the 3x12 inch N.A.S.A. The two molds provided can be used to mold all three. The 4x4 inch mold handles only one 3x3 inch edge crack specimen, while the 4x13 inch mold accommodates either three 3x3 inch specimen, four 3/4x4 1/2 inch tensile specimen or one 3x12 inch N.A.S.A. specimen. Also it will accommodate one 3x3 inch edge crack specimen with two 3/4x4 1/2 inch tensile specimen by using the "tensile spacers" on top of the tensile specimen before the top of the mold is placed into position.

Regardless of the shape, each specimen is made in the manner described in the following paragraphs. First put the bolts through the bottom half of the mold as shown in Figure 7. Spray both halves of the mold with oven coating

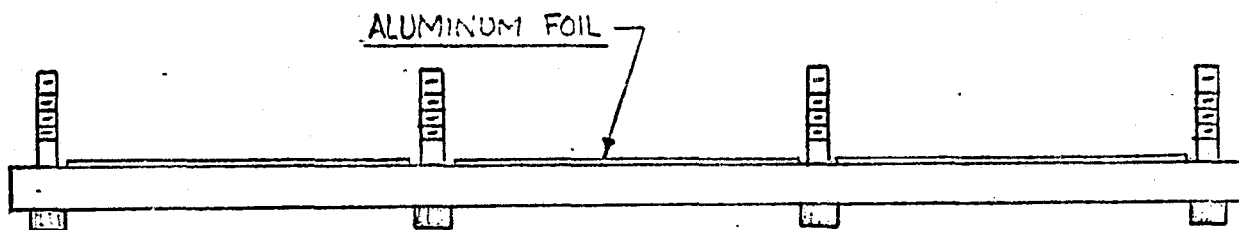


Figure 7.

to prevent sticking. Cut pieces of aluminum foil slightly larger than the specimen size and place one piece with the shiny side up on the bottom part of the mold at each location where the specimens are to be built. At this point if the 3x3 specimens are being made, drop the .060 in. thick spacers over each bolt to insure a uniformly thick specimen. The other specimens do not require such spacers. Now stack the cut pieces according to the specification given at the end of this paper. Once the specimens are stacked, place another piece of aluminum foil on the top of the stack with the dull side showing. Label each specimen with a marking pen (include % coating, volume fraction, fiber lay direction and data molded). Place the top plate of the mold into position and fasten in the manner shown in Figure 8.

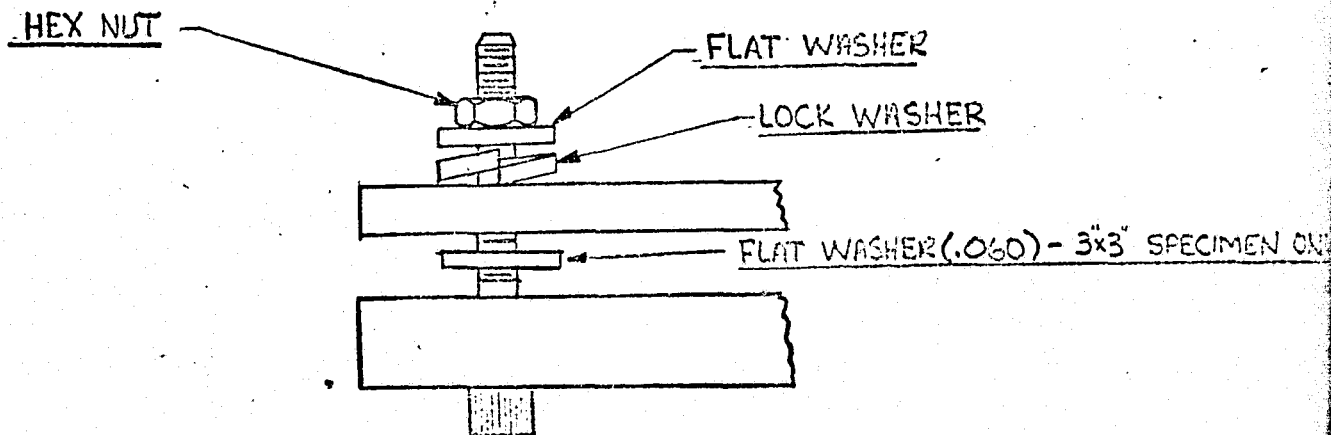


Figure 8.

Tighten the nuts as evenly as possible. Place the entire mold in the roaster for 12 hours at a temperature of 120°C. To do this set the clock on the automatic timer directly opposite the two adjacent pins and turn the switch in the back fully clockwise to turn the timer and roaster on. The roaster temperature setting for 120°C is approximately at the second black mark on the temperature dial, but large variations in this point make it necessary to monitor the temperature with a thermometer. The timer will automatically turn the roaster off after baking the specimen 12 hours and will subsequently turn the roaster on every 24 hours for 20 minute periods. This re-baking does not harm the specimen so that the specimen may be left in the roaster over the weekend for convenience.

Specimen Finishing

After the molding, the aluminum foil may be left on during finishing of the specimen. At this point the N.A.S.A. specimens are completed and are ready to be shipped. However, the others require further work.

The 3x3 inch edge crack specimen must be prepared first by sanding the edges smooth on a belt sander. The edges must be sanded until the finished piece is approximately the same size as the "model piece" (This piece should be marked). Then place the model and sanded specimen together and mark the hole and crack positions. A felt pen works well for determining the hole positions and a razor blade run in the crack of the model works well in marking the crack position. The holes are then punched in the piece with the use of a hammer and special punch and die. To make the starting crack, hold the specimen in the vise and use a hack-saw to cut the crack to the desired length. Due to extreme wear conditions during the cutting of these specimens, many hack-saw blades are needed. The 3x3 inch specimens are now ready for testing.

In finishing the tensile specimens, one must use a belt sander to establish a reference surface on all the pieces before any further work can be done. Also, sand the ends of the tensile bars as shown in Figure 9.

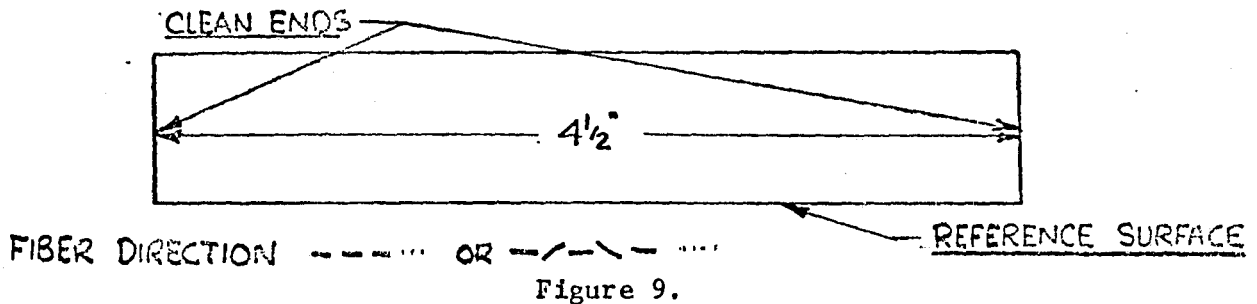


Figure 9.

Once the reference surface is established, scribe the following pattern onto one of the pieces as shown in Figure 10. Start all measurements at the reference surface. With this pattern as the front piece, stack 5 or 6 tensile

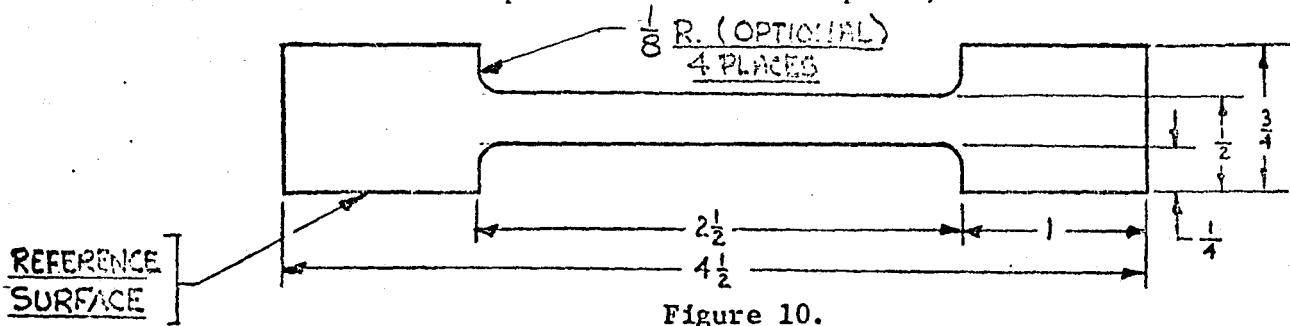


Figure 10.

specimens together so that they may be machined simultaneously with a grinder in the machine shop. At least 4 pieces must be done at once to insure sufficient stiffness in the workpiece during grinding. For the details of the grinding operation see Mr. Clarence Johnson in the machine shop. Once the grinding is complete the specimens are complete. Note: The radius indicated, is optional and also that no holes are punched in the tensile specimen.

In closing it might be said that the order of some of the steps given here are subject to personal preference only, and so each operator may want to modify the procedure as he sees fit.

COATING CONTROL SETTINGS*

Volume fraction = .20 - .25

Drum motor setting = 60 volts D.C.

Repeat Distance (inches)	Coated Length (inches)	Uncoated Length (inches)	Rate (X 100)	Width (X 100)
1	.625	.375	980	285
1	.500	.500	980	335
1	.375	.625	980	410
1	.250	.750	980	500
1	.750	.250	980	150
1	.125	.875	980	550
1	.875	.125	980	065
2	.250	1.750	870	100
2	1.000	1.000	870	735

Volume fraction = .45 - .50

Drum motor setting = 145 volts D.C.

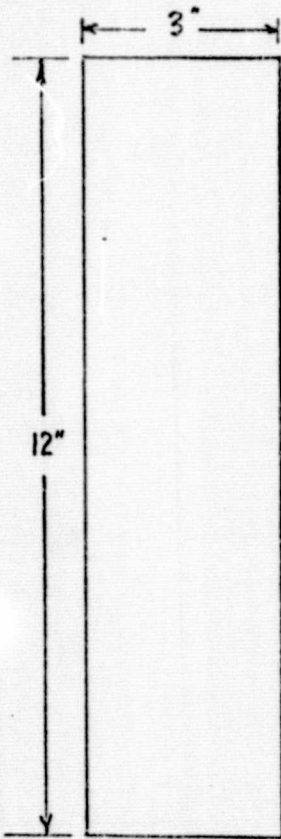
Repeat Distance (inches)	Coated Length (inches)	Uncoated Length (inches)	Rate (X 100)	Width (X 100)
1	.500	.500	995	240
1	.750	.250	995	110
2	1.750	.250	975	650

* For these settings to be correct the air pressure must be set between 40 and 50 psig.

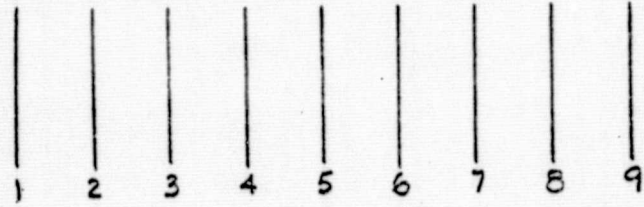
Note:

1. These settings are only approximate and require adjustment before and during each Composite tape making operation.
2. Additional settings are in the laboratory notebook (Winter - 1975).

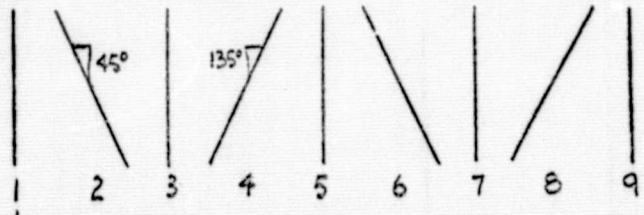
9 - PLY (NASH PANELS) $V_f = .45 - .50$



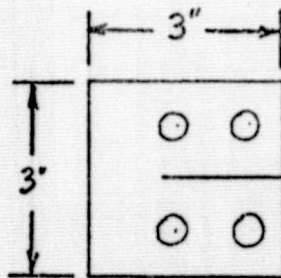
FIBER DIRECTION FOR UNIDIRECTIONAL SPECIMEN



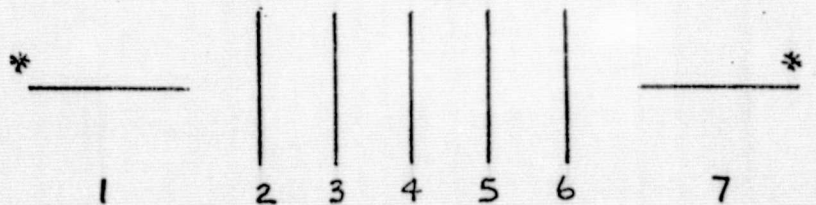
FIBER DIRECTION FOR CROSS-PLY SPECIMEN (0° - 45° - 90°)



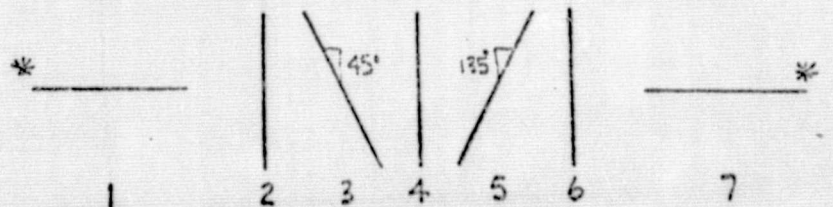
5 - PLY (UNIVERSITY OF MICHIGAN PANELS) $V_f = .20 - .25$



FIBER DIRECTION FOR UNIDIRECTIONAL SPECIMEN



FIBER DIRECTION FOR CROSS-PLY SPECIMEN (0° - 45° - 90°)



* REINFORCING LAYERS